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=> set plurals on perm  
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FILE COVERS 1907 - 1 Dec 2003 VOL 139 ISS 23

FILE LAST UPDATED: 30 Nov 2003 (20031130/ED)

This file contains CAS Registry Numbers for easy and accurate substance identification.

=> e charpentier/au

E1	1	CHARPENTIE YUAN/AU
E2	3	CHARPENTIE YVAN/AU
E3	5 -->	CHARPENTIER/AU
E4	7	CHARPENTIER A/AU
E5	1	CHARPENTIER AGNES/AU
E6	2	CHARPENTIER ALAIN/AU
E7	1	CHARPENTIER ALAIN YVES PIERRE/AU
E8	3	CHARPENTIER ALICE/AU
E9	1	CHARPENTIER ALLAN MATUTE/AU
E10	1	CHARPENTIER ANNE/AU
E11	6	CHARPENTIER APRIL/AU
E12	1	CHARPENTIER APRIL H/AU

=> s e3

L1 5 CHARPENTIER/AU

=> d l1 1-5 ibib abs

L1 ANSWER 1 OF 5 CAPLUS COPYRIGHT 2003 ACS on STN

ACCESSION NUMBER: 1933:48958 CAPLUS

DOCUMENT NUMBER: 27:48958

ORIGINAL REFERENCE NO.: 27:4384e-f

TITLE: Gasoline

AUTHOR(S): **Charpentier**

SOURCE: ATZ, Automobiltechnische Zeitschrift (1933), 36, 126-7  
CODEN: AUTZA6; ISSN: 0001-2785

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

AB The antiknock properties of gasoline do not depend on the class of hydrocarbons of which the gasoline is composed. Thus a gasoline contg. a high proportion of satd. hydrocarbons with side chains may have better antiknock properties than a gasoline with a high content of olefins. The diln. of crank-case oil depends on the oil itself and the gasoline.

L1 ANSWER 2 OF 5 CAPLUS COPYRIGHT 2003 ACS on STN

ACCESSION NUMBER: 1932:17894 CAPLUS  
DOCUMENT NUMBER: 26:17894  
ORIGINAL REFERENCE NO.: 26:1870d-e  
TITLE: The crystalline sodium salt of .alpha.-glycerophosphate  
AUTHOR(S): **Charpentier**; Bocquet  
SOURCE: Compt. rend. (1932), 194, 104-7  
DOCUMENT TYPE: Journal  
LANGUAGE: Unavailable

AB The .alpha.-glycerophosphate of Na, contrary to past belief, crystallizes in octahedral hydrated (6H<sub>2</sub>O) crystals which are neither deliquescent nor efflorescent. The glycerophosphoric acid obtained from the strychnine salt has [.alpha.]<sub>D</sub> = -0.5.degree.. The tetramethyl ether of this acid has [.alpha.]<sub>D</sub> = +5.1.degree.. On sapon. of this ether the resulting dimethylated Na salt has [.alpha.]<sub>D</sub> = -7.2.degree..

L1 ANSWER 3 OF 5 CAPLUS COPYRIGHT 2003 ACS on STN

ACCESSION NUMBER: 1932:16553 CAPLUS  
DOCUMENT NUMBER: 26:16553  
ORIGINAL REFERENCE NO.: 26:1749a  
TITLE: Recent experiences and opinions on automobile and airplane fuels  
AUTHOR(S): **Charpentier**  
SOURCE: ATZ, Automobiltechnische Zeitschrift (1932), 35, 14-5  
CODEN: AUTZA6; ISSN: 0001-2785  
DOCUMENT TYPE: Journal  
LANGUAGE: Unavailable

AB A general discussion on the use of alc-gasolinebenzene mixts. and the individual components in internal-combustion engines.

L1 ANSWER 4 OF 5 CAPLUS COPYRIGHT 2003 ACS on STN

ACCESSION NUMBER: 1916:5387 CAPLUS  
DOCUMENT NUMBER: 10:5387  
ORIGINAL REFERENCE NO.: 10:1050c-e  
TITLE: The tubercle bacillus and arsenic  
AUTHOR(S): **Charpentier**  
CORPORATE SOURCE: Paris  
SOURCE: Ann. inst. Pasteur (1915), 29, 443-58  
DOCUMENT TYPE: Journal  
LANGUAGE: Unavailable

AB The tubercle bacillus grows well in a bouillon containing 1/675, of its wt. of As in the form of Na arsenate. To retard the growth of the organism double this concn. was necessary. The organism likewise grows well in the presence of atoxyl, a little less actively in the presence of Na methylarsonate and with difficulty in the presence of Na cacodylate. The organisms themselves were found to take up As from these media containing arsenical compds. Such organisms were just as virulent for guinea pigs as those grown on the usual media. In studying the action of the same compds. upon other organisms, yeast and aspergillus were found to be unaffected in the same degree as the tubercle bacillus. Injections of Na. cacodylate do not modify at all the course of tuberculous infection in guinea pigs inoculated with very virulent strains although the treatment is begun at once.

L1 ANSWER 5 OF 5 CAPLUS COPYRIGHT 2003 ACS on STN

ACCESSION NUMBER: 1912:23852 CAPLUS  
DOCUMENT NUMBER: 6:23852  
ORIGINAL REFERENCE NO.: 6:3338d-e  
TITLE: Study of the Filter Press Cake of a Sugar Factory  
AUTHOR(S): Lindet, L.; **Charpentier**  
SOURCE: Orig. Com. 8th Intern. Congr. Appl. Chem. (1912), 8,

31-2

DOCUMENT TYPE: Journal  
LANGUAGE: Unavailable

AB ACOH is to be preferred to phenol to dissolve the trisaccharate of lime in detg. this constituent in filter cake, for the phenol scarcely dissolves more than pure H<sub>2</sub>O does, i. e., only the free sugar and the mono- and di-basic saccharates of lime. The phenol can, however, decompose the compds. of CaO with proteins and pectic acid. The cake, with properly prepared and washed with ammoniacal liquor, does not contain free lime, and if, because of imperfect washing, it contains sugar, it is free sugar and not sol. saccharates. The cake contains more or less trisaccharate of lime, the more, the less completely the CaO was slaked and the more rapid the carbonatation.

=> s e4

L2 7 "CHARPENTIER A"/AU

=> d 12 1-7 ibib abs

L2 ANSWER 1 OF 7 CAPLUS COPYRIGHT 2003 ACS on STN

ACCESSION NUMBER: 2002:460965 CAPLUS

DOCUMENT NUMBER: 137:87899

TITLE: Fumagillin treatment of intestinal microsporidiosis

AUTHOR(S): Molina, Jean-Michel; Tourneur, Muriel; Sarafti, Claudine; Chevret, Sylvie; de Gouvello, Amaury; Gobert, Jean-Gerard; Balkan, Suna; Derouin, Francis; Brousse, Paul; Merad, M.; Vittecoq, D.; Duclos-Vallee, J.-C.; Saliba, F.; **Charpentier, A.**; Rudant, E.; Jeanblanc, F.; Treille-Ritouet, D.; Nageotte, A.; de Monbrison, F.; Rabodonorina, M.; Maiza, H.; Pouteil-Noble, C.; Goedert, G.; Felman, P.; Lebrun, J. P.; Perret, C.; Cabrit, R.; Beguinot, I.; Remy, G.; Daliphard, S.; Gourdier, B.; Gerard, L.; Clauvel, J. P.; Noguera, M.-E.; Sigaux, F.

CORPORATE SOURCE: Agence Nationale de Recherches sur le SIDA 090 Study Group, Dep. Infectious Diseases, Hopital Saint-Louis, Univ. Paris VII, Paris, 75475, Fr.

SOURCE: New England Journal of Medicine (2002), 346(25), 1963-1969

CODEN: NEJMAG; ISSN: 0028-4793

PUBLISHER: Massachusetts Medical Society

DOCUMENT TYPE: Journal

LANGUAGE: English

AB Intestinal microsporidiosis due to *Enterocytozoon bienersi* is a cause of chronic diarrhea, malabsorption, and wasting in immunocompromised patients. Currently, there is no effective treatment. We conducted a randomized, double-blind, placebo-controlled trial of fumagillin (60 mg per day orally for two weeks) in patients with chronic *E. bienersi* infection. Efficacy was assessed primarily by the clearance of microsporidia, as evidenced by anal. of stool specimens. Patients in whom microsporidia were not cleared received treatment for two weeks with open-label fumagillin. After clearance of the parasite, follow-up stool exams. were performed monthly to detect relapses. Twelve patients were enrolled in this study, 10 with the acquired immunodeficiency syndrome and 2 who had received organ transplants. Clearance of microsporidia occurred in all six of the patients in the fumagillin group, as compared with none of the six in the placebo group ( $P = 0.002$ ). Treatment with fumagillin was also assocd. with increases in absorption of D-xylose ( $P = 0.003$ ) and in Karnofsky performance scores ( $P < 0.001$ ) and with decreases in loperamide use ( $P = 0.01$ ) and total stool wt. ( $P = 0.04$ ). There were serious adverse events (neutropenia and thrombocytopenia) in three patients in the

fumagillin group; one patient in the placebo group had severe diarrhea. All six controls subsequently had clearance of microsporidia after open-label treatment with fumagillin. Relapses of the infection were identified in two patients during follow-up (median follow-up, 10 mo). Fumagillin is an effective treatment for chronic *E. bienersi* infection in immunocompromised patients.

REFERENCE COUNT: 32 THERE ARE 32 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L2 ANSWER 2 OF 7 CAPLUS COPYRIGHT 2003 ACS on STN

ACCESSION NUMBER: 2000:172819 CAPLUS  
DOCUMENT NUMBER: 133:236174  
TITLE: Nucleotide metabolism and ADPase activity in cardiac and renal transplantation  
AUTHOR(S): Caillard, S.; **Charpentier, A.**; Ravannat, C.; Cassel, D.; Wiesel, M. L.; Moulin, B.; Cazenave, J. P.; Gachet, C.  
CORPORATE SOURCE: Inserm U311 and Service de Nephrologie, Hopitaux Universitaires de Strasbourg, Strasbourg, Fr.  
SOURCE: Transplantation Proceedings (2000), 32(2), 466-467  
CODEN: TRPPA8; ISSN: 0041-1345  
PUBLISHER: Elsevier Science Inc.  
DOCUMENT TYPE: Journal  
LANGUAGE: English

AB ADPase in cardiac and renal transplant recipients was investigated by quantifying the presence of differentiation marker CD39 on lymphocytes and by measuring ADPase enzymic activity on platelets and in plasma. Endothelial activation was studied by detg. the following factors liberated by the endothelium: von Willebrand factor, tissue-plasminogen activator (t-PA), and plasminogen activator inhibitor-1 (PAI-1). A decrease in the no. lymphocytes expressing CD39 was obsd. in transplant patients. This decrease in lymphocytes expressing CD39 can be linked to B lymphocytopenia because CD39 is mainly a B-cell marker. Nonetheless, the enzymic function of CD39 was not explored on lymphocytes. Moreover, the part of the enzymic activity of lymphocytic ectonucleotidase is minimal compared to that in whole blood, given lymphocytopenia affects in most transplant recipients. A redn. in plasma enzyme activity in grafted patients was obsd. Such a difference was not found in washed platelets. This decrease of ADPase activity could be integrated in the general context of ectonucleotidase inhibition. ADPase activity of platelet-rich plasma is about four times higher than that of washed platelets, suggesting a plasmatic ADPase activity. Finally, there was a substantial increase in PAI-1 in transplant patients, indicating a change in the fibrinolytic system, secondary to endothelial aggression in transplant recipients.

REFERENCE COUNT: 11 THERE ARE 11 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L2 ANSWER 3 OF 7 CAPLUS COPYRIGHT 2003 ACS on STN

ACCESSION NUMBER: 1998:582552 CAPLUS  
DOCUMENT NUMBER: 129:342247  
TITLE: Vascular and neuroendocrine components in altered blood pressure regulation after surgical repair of coarctation of the aorta  
AUTHOR(S): Roegel, J-C.; Heinrich, E.; De Jong, W.; Stephan, D.; **Charpentier, A.**; Eisenmann, B.; Imbs, J-L.  
CORPORATE SOURCE: Institut de Pharmacologie, Faculte de Medecine, Strasbourg, Fr.  
SOURCE: Journal of Human Hypertension (1998), 12(8), 517-525  
CODEN: JHHYEN; ISSN: 0950-9240  
PUBLISHER: Stockton Press  
DOCUMENT TYPE: Journal

LANGUAGE: English

AB To investigate potential vascular and neuroendocrine determinants of altered blood pressure (BP) regulation in patients previously operated on for aortic coarctation. We prospectively re-evaluated 45 patients operated on for aortic coarctation at Strasbourg University Hospital over a 13-yr period. Four of these patients were less than 2 yr old at the time of the operation and four were older than 20 yr. Patient age and time since the operation were on av. 21 yr and 8 yr, resp. Surgery consisted of a resection with end-to-end anastomosis for 18 patients, angioplasty, prosthesis or subclavian flap. Despite repair of the coarctation, about 40% of the patients showed an abnormal BP status at rest. The majority of these patients had uncomplicated borderline hypertension. The orthostasis test as well as the BP circadian rhythm were frequently abnormal. While the ankle/arm systolic pressure index measured at rest was generally within the normal range, diminished carotid-femoral pulse wave velocity was obsd. Plasma adrenaline and aldosterone levels were elevated in about 50% of the patients examd. These new findings suggest that there are "cause and effect" relationships between aortic structural and functional vascular abnormalities, and augmented plasma adrenaline and aldosterone in some patients after coarctation repair. These phenomena are likely to be involved in altered BP regulation and might result in recurrent hypertension.

REFERENCE COUNT: 32 THERE ARE 32 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L2 ANSWER 4 OF 7 CAPLUS COPYRIGHT 2003 ACS on STN

ACCESSION NUMBER: 1992:431149 CAPLUS

DOCUMENT NUMBER: 117:31149

TITLE: Black organomineral finishing. Technical factors of success

AUTHOR(S): Raulin, F.; **Charpentier, A.**

CORPORATE SOURCE: Div. Trait. Electrolytiques, Soc. Cont. Parker, Fr.

SOURCE: Galvano-Organo (1992), 61(622), 21-4

CODEN: GAOREN; ISSN: 0302-6477

DOCUMENT TYPE: Journal

LANGUAGE: French

AB Zn plating in an alk. bath, chromating, and impregnation with an organomineral coating are described. Treatment of various effluents is outlined.

L2 ANSWER 5 OF 7 CAPLUS COPYRIGHT 2003 ACS on STN

ACCESSION NUMBER: 1987:417050 CAPLUS

DOCUMENT NUMBER: 107:17050

TITLE: Supposed mechanisms of action of amphetamines on behavior

AUTHOR(S): Bourin, M.; **Charpentier, A.**

CORPORATE SOURCE: Psychopharmacologie Chu Nantes, Nantes, 44035, Fr.

SOURCE: Journal de Psychiatrie Biologique et Therapeutique (1986), 23, 4-8

CODEN: JPBTEY; ISSN: 0292-2509

DOCUMENT TYPE: Journal; General Review

LANGUAGE: French

AB A review, with 14 refs., correlating the biochem. effects of amphetamine with its actions on locomotor, stereotyped, circling, and other types of behavior.

L2 ANSWER 6 OF 7 CAPLUS COPYRIGHT 2003 ACS on STN

ACCESSION NUMBER: 1979:59852 CAPLUS

DOCUMENT NUMBER: 90:59852

TITLE: Creep measurement of sialon sintered without additive

AUTHOR(S): Cabannes, F.; **Charpentier, A.**

CORPORATE SOURCE: Cent. Rec. Phys. Hautes Temp., Univ. Orleans, Orleans,

Fr.  
SOURCE: Science of Ceramics (1977), 9, 510-18  
CODEN: SCCEAW; ISSN: 0080-7575  
DOCUMENT TYPE: Journal  
LANGUAGE: English  
AB High temp. compressive creep of hot-pressed sialon materials without additives and rich in Al<sub>2</sub>O<sub>3</sub> (Al:Si ratio .apprx.1) was studied. The sialon had creep activation energy 158 .+- . 15 kcal/mol and power-law stress dependence with n = 1.9 .+- . 0.2 owing to grain boundary sliding. A high creep rate was due to the ultrafineness of the grains (0.2-0.5 .mu.).

L2 ANSWER 7 OF 7 CAPLUS COPYRIGHT 2003 ACS on STN  
ACCESSION NUMBER: 1910:3265 CAPLUS  
DOCUMENT NUMBER: 4:3265  
ORIGINAL REFERENCE NO.: 4:550g  
TITLE: A Method of Preparing Colloidal Mercury  
AUTHOR(S): **Charpentier, A.**; Guilloz, Th.  
SOURCE: Comptes Rendus des Seances de la Societe de Biologie et de Ses Filiales (1910), 66, 679-80  
CODEN: CRSBAW; ISSN: 0037-9026  
DOCUMENT TYPE: Journal  
LANGUAGE: Unavailable  
AB Unavailable

=> e p a charpentier/au

E1	13	P/AU
E2	1	P A BAEZ/AU
E3	0 -->	P A CHARPENTIER/AU
E4	1	P A MOHAMED NAJAR/AU
E5	1	P A SERMON/AU
E6	2	P A VALLENAS/AU
E7	1	P ACHER R/AU
E8	4	P ACK KIM SUN/AU
E9	1	P AGANI ARIANA P/AU
E10	1	P ALBARSANZ M A/AU
E11	1	P ALBERTO MANRIQUE/AU
E12	1	P ALBERTO MULLER/AU

=> s p. a. charpentier/au

L3 0 P. A. CHARPENTIER/AU

=> e desimone/au

E1	1	DESIMO MARTIN/AU
E2	1	DESIMON LASZLO B/AU
E3	0 -->	DESIMONE/AU
E4	6	DESIMONE A/AU
E5	2	DESIMONE A F/AU
E6	1	DESIMONE A P/AU
E7	1	DESIMONE ANDREW/AU
E8	1	DESIMONE ANTOINETTE/AU
E9	10	DESIMONE ANTONIO/AU
E10	1	DESIMONE ARTHUR R/AU
E11	1	DESIMONE C/AU
E12	1	DESIMONE CARLOS A/AU

=> e j m desimone/au

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E3	0 -->	J M DESIMONE/AU
E4	1	J M T/AU

E5	1	J MAL ANIS/AU
E6	1	J N Z/AU
E7	1	J NAME NOT TRANSLATED/AU
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E9	1	J P/AU
E10	1	J P F/AU
E11	1	J P SCHIFFER J P/AU
E12	1	J R/AU

=> e desimone j m/au

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E3	201 -->	DESIMONE J M/AU
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E5	45	DESIMONE JOHN A/AU
E6	2	DESIMONE JOHN N/AU
E7	54	DESIMONE JOSEPH/AU
E8	1	DESIMONE JOSEPH A/AU
E9	310	DESIMONE JOSEPH M/AU
E10	2	DESIMONE JOSEPH MARK/AU
E11	1	DESIMONE JOSEPH P/AU
E12	1	DESIMONE LAURA/AU

=> e charpentier p a/au

E1	1	CHARPENTIER NOELLE/AU
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E4	3	CHARPENTIER P G/AU
E5	5	CHARPENTIER P L/AU
E6	1	CHARPENTIER PASCALE/AU
E7	6	CHARPENTIER PATRICIA/AU
E8	29	CHARPENTIER PAUL/AU
E9	9	CHARPENTIER PAUL A/AU
E10	1	CHARPENTIER PAUL ABRAHAMS/AU
E11	130	CHARPENTIER PH/AU
E12	8	CHARPENTIER PHILIPPE/AU

=> s e3

L4 4 "CHARPENTIER P A"/AU

=> d l4 1-4 ibib abs

L4 ANSWER 1 OF 4 CAPLUS COPYRIGHT 2003 ACS on STN

ACCESSION NUMBER: 2000:176303 CAPLUS

DOCUMENT NUMBER: 132:308708

TITLE: Long-chain branching in slurry polymerization of ethylene with zirconocene dichloride/modified methylaluminoxane

AUTHOR(S): Kolodka, E.; Wang, W.-J.; Charpentier, P. A.; Zhu, S.; Hamielec, A. E.

CORPORATE SOURCE: Department of Chemical Engineering, McMaster University, Hamilton, ON, L8S 4L7, Can.

SOURCE: Polymer (2000), 41(11), 3985-3991

CODEN: POLMAG; ISSN: 0032-3861

PUBLISHER: Elsevier Science Ltd.

DOCUMENT TYPE: Journal

LANGUAGE: English

AB An exptl. study was carried out of long chain branching (LCB) in ethylene slurry polymn. with bis(cyclopentadienyl) zirconium dichloride (Cp2ZrCl2)/modified methylaluminoxane (MMAO, MAO contg. 12% isobutylaluminoxane) using a semi-batch reactor. The effects of reaction temp., pressure, MMAO concn., and catalyst feeding method on the long



chain branching d. (LCBD, no. of branches per 10 000 carbons), polymer mol. wt., and shear thinning property (I10/I2) were systematically examd. The slurry polymn. process, with its assocd. polymer-rich phase and the partitioning of active sites, favors the LCB formation via an in situ copolymn. of ethylene macromonomers generated by .beta.-hydride elimination and chain transfer to monomer. Increasing the temp. from 60 to 80.degree. reduced the LCBD from 0.33 to 0.10, while increasing the pressure from 2 to 20 psig reduced the LCBD from 0.73 to 0.30. The LCB polyethylenes showed enhanced shear thinning properties, with melt flow index ratio (I10/I2) of 8.8-21.5. The feeding sequence of reactants also had a significant effect on the LCB formation. Feeding ethylene monomer before zirconocene catalyst produced polyethylenes having much higher LCBD than feeding catalyst before monomer.

REFERENCE COUNT: 22 THERE ARE 22 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 2 OF 4 CAPLUS COPYRIGHT 2003 ACS on STN

ACCESSION NUMBER: 1999:505233 CAPLUS  
DOCUMENT NUMBER: 131:272213  
TITLE: Continuous Polymerizations in Supercritical Carbon Dioxide: Chain-Growth Precipitation Polymerizations  
AUTHOR(S): Charpentier, P. A.; Kennedy, K. A.; DeSimone, J. M.; Roberts, G. W.  
CORPORATE SOURCE: Department of Chemical Engineering, North Carolina State University, Raleigh, NC, 27695-7905, USA  
SOURCE: Macromolecules (1999), 32(18), 5973-5975  
CODEN: MAMOBX; ISSN: 0024-9297  
PUBLISHER: American Chemical Society  
DOCUMENT TYPE: Journal  
LANGUAGE: English

AB The feasibility of the continuous pptn. polymn. of vinylidene fluoride (I) was demonstrated using an intensely agitated continuous stirred tank reactor (CSTR). Rates of polymn. of I in CSTR were significantly higher than the av. rates of batch polymn. under similar conditions. This represented the completion of the first stage of a three-stage project. The second stage, which was in progress, involved continuous removal of the polymer powder from the high-pressure filters. An objective of this stage was to minimize the pressure drop of the supercrit. fluid/monomer mixt. in order to minimize the energy required to recompress the mixt. for recycle. The third stage of this project involved recycle of both monomer and supercrit. CO2.

REFERENCE COUNT: 7 THERE ARE 7 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 3 OF 4 CAPLUS COPYRIGHT 2003 ACS on STN

ACCESSION NUMBER: 1998:682936 CAPLUS  
DOCUMENT NUMBER: 129:316612  
TITLE: Effect of aluminoxane on semi-batch polymerization of ethylene using zirconocene dichloride  
AUTHOR(S): Charpentier, P. A.; Zhu, S.; Hamielec, A. E.; Brook, M. A.  
CORPORATE SOURCE: Dep. Chem. Eng., McMaster Univ., Hamilton, ON, L8S 4L7, Can.  
SOURCE: Polymer (1998), 39(25), 6501-6511  
CODEN: POLMAG; ISSN: 0032-3861  
PUBLISHER: Elsevier Science Ltd.  
DOCUMENT TYPE: Journal  
LANGUAGE: English

AB Several types of com. and developmental aluminoxanes were studied in the semi-batch polymn. of ethylene using bis(cyclopentadienyl) zirconium dichloride (Cp2ZrCl2). High temp. gel permeation chromatog. (GPC) measurements of polyethylene (PE) formed using various types of

aluminoxanes approximated Glory's most probable distribution from a single site-type with PE Mw/Mns approaching 2.0 for polymn. temps. ranging from 50 to 90.degree.C. The structure and type of aluminoxane co-catalyst were found not to influence the mol. wt. distribution (MWD) of PE although to influence the activity of the Cp2ZrCl2 catalyst. In general, modified methylaluminoxanes (MMAOs) and methylaluminoxanes (MAOs) were found to have similar activities, whereas isobutylaluminoxane gave a very low activity in the polymn. of ethylene. Increasing iso-Bu content in the aluminoxane co-catalyst led to a continual decrease in catalyst activity. Increasing Al/Zr molar ratio, by increasing the MAO concn., led to increasing catalyst activity up to a max. activity at an Al/Zr molar ratio of 2400. Like the structure and type of the aluminoxane co-catalyst, the Al/Zr molar ratio was found not to influence the PE MWD. Mixing in addnl. trimethylaluminum with MAO was found not to alter the MWD but gave lower activity at similar Al/Zr molar ratios than that from MAO itself.

REFERENCE COUNT: 47 THERE ARE 47 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 4 OF 4 CAPLUS COPYRIGHT 2003 ACS on STN

ACCESSION NUMBER: 1997:716258 CAPLUS  
DOCUMENT NUMBER: 127:307721  
TITLE: Continuous solution polymerization of ethylene using metallocene catalyst system zirconocene dichloride/metallocene-methylaluminoxane/trimethylaluminum  
AUTHOR(S): Charpentier, P. A.; Zhu, S.; Hamielec, A. E.; Brook, Michael A.  
CORPORATE SOURCE: Departments of Chemical Engineering and Chemistry, McMaster University, Hamilton, ON, L8S 4L7, Can.  
SOURCE: Industrial & Engineering Chemistry Research (1997), 36(12), 5074-5082  
CODEN: IECRED; ISSN: 0888-5885  
PUBLISHER: American Chemical Society  
DOCUMENT TYPE: Journal  
LANGUAGE: English

AB A high-pressure, high-temp. continuous-stirred-tank reactor system having approx. an ideal residence time distribution (RTD) has been set up for the polymn. of ethylene with the metallocene catalyst system zirconocene dichloride (Cp2ZrCl2)-Me aluminoxane-/Me3Al in PhMe at 1500 psig have been carried out. The reactor system showed good control over temp., pressure, and ethylene feed rate. The steady state was obtained after four mean residence times (4 .tau.'s). With increasing Cp2ZrCl2 concn., the mol. wt. (MW) of polyethylene (PE) decreased and the catalyst activity (kg of PE/[ethylene].cntdot.g of catalyst) increased. With increasing temp. between 140 and 200.degree., the MW of PE decreased and mol. wt. distribution polydispersity increased. PEs with wt.-av. mol. wt. (Mw) 18,000-52,000 were obtained. The catalyst activity decreased with increasing temp. with an apparent activation energy of -93 kJ/mol. The deactivation of the catalyst is first-order with the rate const. kd = 2.1 .times. 10-3 s-1 at 140.degree.. The rate consts. of propagation and .beta.-scission at 140.degree. are kp = 5 .times. 103 (M.cntdot.s)-1 and ktr,.beta. = 3 s-1, resp.

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L5 9 "CHARPENTIER PAUL A"/AU

=> d 15 1-9 ibib abs

L5 ANSWER 1 OF 9 CAPLUS COPYRIGHT 2003 ACS on STN

ACCESSION NUMBER: 2002:661536 CAPLUS  
DOCUMENT NUMBER: 137:311291

TITLE: Continuous precipitation polymerization of vinylidene fluoride in supercritical carbon dioxide: formation of polymers with bimodal molecular weight distributions  
AUTHOR(S): Saraf, Manish K.; Gerard, Sylvain; Wojcinski, Louis M., II; **Charpentier, Paul A.**; DeSimone, Joseph M.; Roberts, George W.  
CORPORATE SOURCE: Department of Chemical Engineering, North Carolina State University, Raleigh, NC, 27695-7905, USA  
SOURCE: Macromolecules (2002), 35(21), 7976-7985  
CODEN: MAMOBX; ISSN: 0024-9297  
PUBLISHER: American Chemical Society  
DOCUMENT TYPE: Journal  
LANGUAGE: English

AB The radical polymn. of vinylidene fluoride in supercrit. carbon dioxide was studied in a continuous stirred tank reactor. Expts. were carried out to investigate the effect of inlet monomer concn., temp., av. residence time, and agitation on the polymn. rate, the av. mol. wts., and the mol. wt. distribution of the poly(vinylidene fluoride). A homogeneous kinetic model that includes inhibition due to chain transfer to monomer predicted the polymn. rates reasonably well. However, imperfect mixing, rather than a chem. effect, may have caused the apparent inhibition obsd. at high monomer concns. At inlet monomer concns. greater than about 1.5 M, broad and bimodal mol. wt. distributions were obsd. An extended homogeneous kinetic model that includes chain transfer to polymer predicted the polydispersities reasonably well. This model also predicted a region of inoperability that matched the exptl. results. However, the extended homogeneous model could not account for the bimodal distributions.

REFERENCE COUNT: 26 THERE ARE 26 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L5 ANSWER 2 OF 9 CAPLUS COPYRIGHT 2003 ACS on STN

ACCESSION NUMBER: 2002:603413 CAPLUS  
DOCUMENT NUMBER: 137:263374  
TITLE: Continuous polymerizations in supercritical carbon dioxide  
AUTHOR(S): **Charpentier, Paul A.**; DeSimone, Joseph M.; Roberts, George W.  
CORPORATE SOURCE: Department of Chemical Engineering, North Carolina State University, Raleigh, NC, 27695-7905, USA  
SOURCE: ACS Symposium Series (2002), 819(Clean Solvents: Alternative Media for Chemical Reactions and Processing), 113-135  
CODEN: ACSMC8; ISSN: 0097-6156  
PUBLISHER: American Chemical Society  
DOCUMENT TYPE: Journal  
LANGUAGE: English

AB We have developed a system for the continuous polymn. of various monomers in scCO<sub>2</sub> and the continuous removal of polymer particles from high-pressure to ambient conditions. Expts. have been performed with the surfactant-free pptn. polymn. of vinylidene fluoride (VDF) utilizing di-Et peroxydicarbonate as the free-radical initiator. The PVDF product was collected as a dry, "free-flowing" powder. Tunable bimodal mol. wt (Mw) distributions were achieved by varying the VDF concn. The conversion of VDF in these polymns. ranged from 7 to 26%, and the rate of polymn. (Rp) reached a max. of 27 .times. 10<sup>-5</sup> mol/L.cntdot.s at VDF feed concn. 2.5 mol/L at 75.degree.C. Homogeneous free-radical kinetics provided a good approxn. for Rp in this heterogeneous polymn. The PVDF was characterized by gel permeation chromatog. and melt flow index (MFI), giving Mw's up to 104 kg/mol, and MFI's as low as 3.0 at 230.degree.C.

REFERENCE COUNT: 35 THERE ARE 35 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L5 ANSWER 3 OF 9 CAPLUS COPYRIGHT 2003 ACS on STN

ACCESSION NUMBER: 2002:540890 CAPLUS  
DOCUMENT NUMBER: 137:232984  
TITLE: Continuous precipitation polymerization of vinylidene fluoride in supercritical carbon dioxide: molecular weight distribution  
AUTHOR(S): Saraf, Manish K.; Wojcinski, Louis M., II; Kennedy, Karen A.; Gerard, Sylvain; **Charpentier, Paul A.**; DeSimone, Joseph M.; Roberts, George W.  
CORPORATE SOURCE: Department of Chemical Engineering, North Carolina State University, Raleigh, NC, 27695-7905, USA  
SOURCE: Macromolecular Symposia (2002), 182(3rd IUPAC-Sponsored International Symposium on Free-Radical Polymerization: Kinetics and Mechanism, 2001), 119-129  
CODEN: MSYMEC; ISSN: 1022-1360  
PUBLISHER: Wiley-VCH Verlag GmbH  
DOCUMENT TYPE: Journal  
LANGUAGE: English

AB The surfactant-free pptn. polymn. of vinylidene fluoride in supercrit. carbon dioxide was studied in a continuous stirred autoclave. The polymn. temp. ranged from 65 to 85.degree.C, the av. residence time in the reactor varied from 10 to 50 min., and the pressure was between 210 and 305 bar. Di-Et peroxydicarbonate was used as the initiator. The fractional conversion of monomer varied from 7 to 26%, the no.-av. mol. wt. of the polymer was between about 14,000 and 79,000, and the wt.-av. mol. wt. was between about 21,000 and 700,000. In many cases, the polymer exhibited a bimodal mol.-wt. distribution, esp. at high monomer concns.

REFERENCE COUNT: 22 THERE ARE 22 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L5 ANSWER 4 OF 9 CAPLUS COPYRIGHT 2003 ACS on STN

ACCESSION NUMBER: 2001:868544 CAPLUS  
DOCUMENT NUMBER: 136:6552  
TITLE: Multimodal fluoropolymers and methods of making the same  
INVENTOR(S): Desimore, Joseph M.; Roberts, George W.;  
**Charpentier, Paul A.**  
PATENT ASSIGNEE(S): University of North Carolina at Chapel Hill, USA;  
North Carolina State University  
SOURCE: PCT Int. Appl., 29 pp.  
CODEN: PIXXD2  
DOCUMENT TYPE: Patent  
LANGUAGE: English  
FAMILY ACC. NUM. COUNT: 1  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2001090206	A2	20011129	WO 2001-US16252	20010518
WO 2001090206	A3	20020620		
W: CN, JP				
RW: AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR				
US 2002040118	A1	20020404	US 2001-860413	20010518
EP 1297032	A2	20030402	EP 2001-941518	20010518
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, FI, CY, TR				
JP 2003534416	T2	20031118	JP 2001-587016	20010518
PRIORITY APPLN. INFO.:				
				US 2000-205817P P 20000519
				WO 2001-US16252 W 20010518

AB A method for forming a fluoropolymer comprises providing a reaction mixt.

comprising carbon dioxide, at least one fluoromonomer (e.g., vinylidene fluoride), and an initiator; and reacting the at least one fluoromonomer in the reaction mixt. to form a fluoropolymer. The fluoropolymer has a multimodal mol. wt. distribution.

L5 ANSWER 5 OF 9 CAPLUS COPYRIGHT 2003 ACS on STN  
ACCESSION NUMBER: 2001:360058 CAPLUS  
DOCUMENT NUMBER: 134:367383  
TITLE: Continuous process for making polymers in carbon dioxide  
INVENTOR(S): **Charpentier, Paul A.**; Desimone, Joseph M.; Roberts, George W.  
PATENT ASSIGNEE(S): North Carolina State University, USA; University of North Carolina at Chapel Hill  
SOURCE: PCT Int. Appl., 58 pp.  
CODEN: PIXXD2  
DOCUMENT TYPE: Patent  
LANGUAGE: English  
FAMILY ACC. NUM. COUNT: 2  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2001034667	A1	20010517	WO 2000-US30765	20001110
W: DE, ES, GB, IL, JP, LU, NO, PL, RO, RU, ZA				
RW: AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR				
EP 1248807	A1	20021016	EP 2000-978446	20001110
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, FI, RO, CY, TR				
JP 2003514083	T2	20030415	JP 2001-537378	20001110
PRIORITY APPLN. INFO.: US 1999-165177P P 19991112				
WO 2000-US30765 W 20001110				

AB A method for carrying out the continuous polymn. of a monomer in a carbon dioxide reaction medium comprises the steps of: (a) providing an app. including a continuous reaction vessel and a separator; (b) carrying out a polymn. reaction in the reaction vessel by combining a monomer and a carbon dioxide reaction medium therein (and preferably by also combining an initiator therein), wherein the reaction medium is a liq. or supercrit. fluid, and wherein the reaction produces a solid polymer product in the reaction vessel; then (c) withdrawing a continuous effluent stream from the reaction vessel during the polymn. reaction, wherein the effluent stream is maintained as a liq. or supercrit. fluid; then (d) passing the continuous effluent stream through the separator and sepg. the solid polymer therefrom while maintaining at least a portion of the effluent stream as a liq. or supercrit. fluid; and then (e) returning at least a portion of the continuous effluent stream to the reaction vessel while maintaining the effluent stream as a liq. or supercrit. fluid. The need for significant recompression of the continuous effluent stream prior to return to the reaction vessel is thereby minimized. App. for carrying out such methods is also disclosed.

REFERENCE COUNT: 6 THERE ARE 6 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L5 ANSWER 6 OF 9 CAPLUS COPYRIGHT 2003 ACS on STN  
ACCESSION NUMBER: 2000:862903 CAPLUS  
DOCUMENT NUMBER: 134:57016  
TITLE: Decomposition of polymerisation initiators in supercritical CO2: a novel approach to reaction kinetics using a CSTR  
AUTHOR(S): **Charpentier, Paul A.**; DeSimone, Joseph M.; Roberts, George W.

CORPORATE SOURCE: Department of Chemical Engineering, Corner of Stinson  
and Current, 113 Riddicks Labs, North Carolina State  
University, Raleigh, NC, 27695-7905, USA  
SOURCE: Chemical Engineering Science (2000), 55(22), 5341-5349  
CODEN: CESCAC; ISSN: 0009-2509  
PUBLISHER: Elsevier Science Ltd.  
DOCUMENT TYPE: Journal  
LANGUAGE: English

AB A novel method for simultaneous detn. of the decompn. rate const.,  $k_D$ , and the initiator efficiency,  $f$ , of a free-radical polymn. initiator has been developed. The decompn. is carried out in a continuous-stirred-tank reactor (CSTR) in the presence of a radical scavenger. Mass balances for the initiator and the scavenger show that both  $f$  and  $k_D$  can be detd. from a set of const.-temp. expts. with a varying reactor mean residence time. Expts. were conducted in supercrit. carbon dioxide (scCO<sub>2</sub>) using di-Et peroxydicarbonate (DEPDC) as the free-radical initiator and galvinoxyl as the free-radical scavenger. The decompn. of DEPDC was first-order between 338 and 358 K. The activation energy,  $E_A$ , was 132 kJ/mol, the pre-exponential factor,  $A_D$ , was  $6.3 \times 10^{16} \text{ s}^{-1}$ , and the initiator efficiency,  $f$ , was 0.6. These values are similar to those reported in the literature for DEPDC decompn. in traditional org. solvents. Varying the carbon dioxide pressure between 240 and 310 bar at 348 K had no effect on the value of  $k_D$ . Transition state theory predicted no significant effect of pressure or solvent compn. on the initiator decompn. rate const.

REFERENCE COUNT: 31 THERE ARE 31 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L5 ANSWER 7 OF 9 CAPLUS COPYRIGHT 2003 ACS on STN

ACCESSION NUMBER: 2000:795324 CAPLUS  
TITLE: Reaction engineering of continuous chain-growth polymerizations in scCO<sub>2</sub>.

AUTHOR(S): **Charpentier, Paul A.**; Roberts, George W.;  
DeSimone, Joseph M.

CORPORATE SOURCE: Kenan CO<sub>2</sub> Center, North Carolina State University,  
Raleigh, NC, 27606, USA

SOURCE: Abstracts of Papers - American Chemical Society  
(2000), 220th, IEC-075  
CODEN: ACSRAL; ISSN: 0065-7727

PUBLISHER: American Chemical Society

DOCUMENT TYPE: Journal; Meeting Abstract

LANGUAGE: English

AB Process, kinetic, and phys./chem. research has been undertaken to demonstrate the feasibility for utilizing scCO<sub>2</sub> technol. for making polymers industrially. An exptl. system has been designed and developed, consisting of a continuous stirred tank reactor (CSTR) for polymn., and a high-pressure filter-withdrawal system where polymer is collected and continuously ejected to ambient conditions. High mol. wt. poly(acrylic acid) and poly(vinylidene fluoride) have been prepd. with this system. A novel initiator decompn. technique has been developed in the CSTR to det. the decompn. kinetics of free-radical initiators in scCO<sub>2</sub>. Transition state theory was successfully applied to predict the exptl. results for initiator decompn. in scCO<sub>2</sub>. Addnl., a kinetic anal. of vinylidene fluoride polymn. has been undertaken in which exptl. data was compared to that predicted from a developed kinetic model. Classical homogeneous kinetics were obtained, illustrating that continuous polymns. in a scCO<sub>2</sub> medium are feasible for this system, and potentially, for prepg. several other industrially important polymers and copolymers.

L5 ANSWER 8 OF 9 CAPLUS COPYRIGHT 2003 ACS on STN

ACCESSION NUMBER: 2000:746369 CAPLUS

DOCUMENT NUMBER: 134:29729

TITLE: Continuous Precipitation Polymerization of Vinylidene

Fluoride in Supercritical Carbon Dioxide: Modeling the Rate of Polymerization

AUTHOR(S): **Charpentier, Paul A.**; DeSimone, Joseph M.; Roberts, George W.  
CORPORATE SOURCE: Department of Chemical Engineering, North Carolina State University, Raleigh, NC, 27695-7905, USA  
SOURCE: Industrial & Engineering Chemistry Research (2000), 39(12), 4588-4596  
CODEN: IECRED; ISSN: 0888-5885  
PUBLISHER: American Chemical Society  
DOCUMENT TYPE: Journal  
LANGUAGE: English

AB The kinetics of the surfactant-free pptn. polymn. of vinylidene fluoride (VF2) in supercrit. carbon dioxide have been studied in a continuous stirred autoclave. Di-Et peroxydicarbonate was used as the free-radical initiator. The stirring rate and agitator design had no effect on the rate of polymn. (Rp) or on the wt.-av. mol. wt. (Mw) of the formed poly(vinylidene fluoride) (PVDF). The fractional conversion of VF2 ranged from 7 to 26%, and Rp was as high as 27 .times. 10<sup>-5</sup> mol/L.cntdot.s at 75 .degree.C and at a VF2 feed concn. of 2.5 mol/L. The PVDF polymer was collected as a dry, "free-flowing" powder and had Mw's up to 150 kg/mol and melt flow indexes as low as 3.0 at 230 .degree.C. Homogeneous, free-radical kinetics provided a reasonable basis for describing the rate of polymn., despite the heterogeneous nature of the system. The order of the reaction with respect to the monomer was found to be 1.0, and the order with respect to the initiator was 0.5. The exptl. data suggest that an inhibitor is present in the monomer or that the monomer itself acts as an inhibitor.

REFERENCE COUNT: 31 THERE ARE 31 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L5 ANSWER 9 OF 9 CAPLUS COPYRIGHT 2003 ACS on STN

ACCESSION NUMBER: 1998:747775 CAPLUS  
DOCUMENT NUMBER: 130:25388  
TITLE: Long-chain branching in ethylene polymerization using constrained geometry metallocene catalyst  
AUTHOR(S): Wang, Wen-Jun; Yan, Dajing; **Charpentier, Paul A.**; Zhu, Shiping; Hamielec, Archie E.; Sayer, Brian G.  
CORPORATE SOURCE: Department Chemical Engineering, McMaster University, Hamilton, ON, L8S 4L7, Can.  
SOURCE: Macromolecular Chemistry and Physics (1998), 199(11), 2409-2416  
CODEN: MCHPES; ISSN: 1022-1352  
PUBLISHER: Wiley-VCH Verlag GmbH  
DOCUMENT TYPE: Journal  
LANGUAGE: English

AB The authors report an exptl. investigation on long chain branching (LCB) in ethylene polymn. with the constrained geometry titanocene catalyst/tris(pentafluorophenyl)boron/modified methylaluminoxane system (CGC-Ti/TPFPB/MMAO) using a continuous stirred-tank reactor (CSTR) at 140.degree., 3.45 .times. 103 kPa, and a mean residence time (.tau.) of 4 min. The effects of the catalyst (CGC-Ti) and co-catalyst (TPFPB and MMAO) concns. on the catalyst activity, polymer mol. wt., and shear thinning were systematically examd. The boron cocatalyst had a great influence on the CGC activity. Increasing the ratio TPFPB/CGC-Ti from 0.66-5 gave ethylene propagation rates from 1.65 .times. 103 to 1.36 .times. 104 L mol<sup>-1</sup> s<sup>-1</sup>. The addn. of MMAO appeared to be essential, most likely acting as an impurity scavenger. The LCB polyethylenes showed enhanced shear thinning properties. The melt flow index ratios I10/I2 were in the range of 6.96-23.4, with the I2 of 0.172-0.681 g/10 min. The wt.-av. mol. wt. .hivin.Mw was correlated to I2 using a power equation

within narrow I10/I2 ranges. The exponential factors were in the range of 4.24-6.31. The exptl. and calcd.  $\ln M_w$  were in a good agreement.  
REFERENCE COUNT: 34 THERE ARE 34 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

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L6 9 "CHARPENTIER PAUL A"/AU

=> s e8  
L7 29 "CHARPENTIER PAUL"/AU

=> d 17 1-29 ibib abs

L7 ANSWER 1 OF 29 CAPLUS COPYRIGHT 2003 ACS on STN  
ACCESSION NUMBER: 2003:381145 CAPLUS  
DOCUMENT NUMBER: 138:338590  
TITLE: The polymerization of fluorinated monomers and the determination of phase equilibria in carbon dioxide  
AUTHOR(S): DeSimone, Joseph M.; Wojcinski, Louis M., III; Kennedy, Karen A.; Zannoni, Luke; Saraf, Manish; Charpentier, Paul; Roberts, George W.  
CORPORATE SOURCE: NSF Cent. for Environ. Responsible Solvents and Processes, Dep. of Chem., Univ. of North Carolina, Chapel Hill, NC, 27599-3290, USA  
SOURCE: Polymeric Materials Science and Engineering (2001), 84, 137  
CODEN: PMSEDG; ISSN: 0743-0515  
PUBLISHER: American Chemical Society  
DOCUMENT TYPE: Journal  
LANGUAGE: English  
AB In our labs, we have focused on the free radical pptn. polymn. of fluorinated monomers, and the scale-up of these reactions in a continuous system. These continuous polymns. have focused on the synthesis of poly(vinylidene fluoride). Using this continuous system, we were able to study the kinetics of these processes, including initiator decompn. and the rate of polymn. Conversions as high as 26%, and  $M_w$ 's as high as 104 kg/mol have been attained using this system. Results are also presented describing the phase equil. of reactant species. These expts., carried out over a range of conditions similar to those used for the polymns., were conducted using a gas chromatograph coupled to an app. built in our labs specifically for these studies. By measuring these phase equil., addnl. insight into the polymn. can be obtained.  
REFERENCE COUNT: 5 THERE ARE 5 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L7 ANSWER 2 OF 29 CAPLUS COPYRIGHT 2003 ACS on STN  
ACCESSION NUMBER: 2002:624894 CAPLUS  
DOCUMENT NUMBER: 137:185909  
TITLE: Polymerization of vinylidene fluoride in dense carbon dioxide  
AUTHOR(S): Wojcinski, Louis M.; Saraf, Manish K.; Charpentier, Paul; DeSimone, Joseph M.; Roberts, George W.  
CORPORATE SOURCE: Dept. of Chem. Eng., North Carolina State Univ., Raleigh, NC, 27605-7905, USA  
SOURCE: Polymer Preprints (American Chemical Society, Division of Polymer Chemistry) (2002), 43(2), 889  
CODEN: ACPPAY; ISSN: 0032-3934  
PUBLISHER: American Chemical Society, Division of Polymer Chemistry  
DOCUMENT TYPE: Journal; (computer optical disk)



LANGUAGE: English

AB Continuous polymn. of vinylidene fluoride in a continuous stirred tank reactor using supercrit. carbon dioxide and initiated by diethylperoxydicarbonate is reported. The PVDF prepd. by this environmentally benign method is highly porous with a novel bimodal mol. wt. distribution.

L7 ANSWER 3 OF 29 CAPLUS COPYRIGHT 2003 ACS on STN

ACCESSION NUMBER: 2001:203772 CAPLUS

TITLE: Polymerization of fluorinated monomers and the determination of phase equilibria in carbon dioxide

AUTHOR(S): DeSimone, Joseph M.; Wojcinski, Louis M., II; Kennedy, Karen A.; Zannoni, Luke; Saraf, Manish; Charpentier, Paul; Roberts, George W.

CORPORATE SOURCE: NSF Center for Environmentally Responsible Solvents and Processes, Department of Chemistry, University of North Carolina, Chapel Hill, NC, 27599-3290, USA

SOURCE: Abstracts of Papers - American Chemical Society (2001), 221st, PMSE-075  
CODEN: ACSRAL; ISSN: 0065-7727

PUBLISHER: American Chemical Society

DOCUMENT TYPE: Journal; Meeting Abstract

LANGUAGE: English

AB In order to harness the advantageous properties of CO<sub>2</sub> for making polymers, new processes are required. In our labs, we have focused on the free radical pptn. polymn. of fluorinated monomers, and the scale-up of these reactions in a continuous system. These continuous polymns. have focused on the synthesis of poly(vinylidene fluoride). Using this continuous system, we have been able to study the kinetics of these processes, including initiator decompn. and the rate of polymn. Conversions as high as 26%, and Mw's as high as 104 kg/mol have been attained using this system. Results are also presented describing the phase equil. of reactant species. These expts., carried out over a range of conditions similar to those used for the polymns., were conducted using a gas chromatograph coupled to an app. built in our labs specifically for these studies. By measuring these phase equil., addnl. insight into the polymn. can be obtained.

L7 ANSWER 4 OF 29 CAPLUS COPYRIGHT 2003 ACS on STN

ACCESSION NUMBER: 1959:40028 CAPLUS

DOCUMENT NUMBER: 53:40028

ORIGINAL REFERENCE NO.: 53:7210d-e

TITLE: Phenothiazines

INVENTOR(S): Charpentier, Paul

PATENT ASSIGNEE(S): Societe des usines chimiques Rhone-Poulenc

SOURCE: 3rd addn. to Fr. 1,075,117 (C.A. 53, 1387g)

DOCUMENT TYPE: Patent

LANGUAGE: Unavailable

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
FR 63114		19550824	FR	

AB ClCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>NMe<sub>2</sub> (8 g.) in 8 g. xylene is added over 1 hr. to a boiling suspension of 13.9 g. bromophenothiazine (I) and 2.5 g. NaNH<sub>2</sub> (80%) in 80 cc. xylene and refluxed 1 hr. Acidified water is added on cooling, the xylene sepd., and the aq. layer basified with NaOH and extd. with Et<sub>2</sub>O to give 10-(3-dimethylaminopropyl)bromophenothiazine, b0.08 186.degree.; HCl salt m. 170-1.degree.. Similarly, the use of MeCHClCH<sub>2</sub>NMe<sub>2</sub> gives a mixt. of 10-(2-dimethylaminopropyl)- and 10-(1-dimethylamino-2-propyl)bromophenothiazine, b0.15 190-2.degree., contg. mainly the former

whose HCl salt m. 203-4.degree. (BuOH). I is obtained by cyclizing m-bromodiphenylamine with S.

L7 ANSWER 5 OF 29 CAPLUS COPYRIGHT 2003 ACS on STN

ACCESSION NUMBER: 1959:23416 CAPLUS  
DOCUMENT NUMBER: 53:23416  
ORIGINAL REFERENCE NO.: 53:4312d-g  
TITLE: Phenothiazines  
INVENTOR(S): **Charpentier, Paul**  
PATENT ASSIGNEE(S): Societe des usines chimiques Rhone-Poulenc  
SOURCE: Addn. to Fr. 1,075,117 (C.A. 53, 1387g)  
DOCUMENT TYPE: Patent  
LANGUAGE: Unavailable  
FAMILY ACC. NUM. COUNT: 1  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
FR 61241		19550405	FR	

AB Further 10-(tertiaryaminoalkyl)chlorophenothiazines are prepd. according to the method of Fr. 1,075,117. Thus, 6 g. Me<sub>2</sub>N(CH<sub>2</sub>)<sub>2</sub>Cl in 6 g. xylene added over 1 hr. to 11.6 g. chlorophenothiazine (I) and 2.4 g. NaNH<sub>2</sub> in 60 cc. boiling xylene, mixt. refluxed further 1 hr., cooled, treated with dil. acid, xylene layer sepd., aq. layer basified with NaOH, base extd. with Et<sub>2</sub>O. Distn. gave 10-(2'-dimethylaminoethyl)chlorophenothiazine b0.9 195.degree., HCl, m. 220-1.degree. (EtOH). In the same way I gave: with Et<sub>2</sub>(CH<sub>2</sub>)<sub>2</sub>Cl, 10-(2'-diethylaminoethyl)chlorophenothiazine, b2.0 215.degree., HCl salt, m. 165-6.degree. (PhCl); with N-(.beta.-chloroethyl)pyrrolidine, 10-(2'-pyrrolidinoethyl)chlorophenothiazine, b1.5 230.degree., HCl salt, m. 164.degree. (PhCl); with Me<sub>2</sub>NCH<sub>2</sub>C(Me)<sub>2</sub>CH<sub>2</sub>Cl, 10-(3'-dimethylamino-2,2'-dimethylpropyl)chlorophenothiazine b3.0 220.degree., HCl salt, m. 182-3.degree. (PhCl); N-(.beta.-chloroethyl)morpholine, 10-(2'-morpholinoethyl)chlorophenothiazine, b2.0 260.degree., HCl salt, m. 192-3.degree. (EtOH); with Et<sub>2</sub>NCH<sub>2</sub>CHClMe, a mixt., b3.0 230-5.degree., contg. mainly 10-(2'-diethylamino-1'-propyl)chlorophenothiazine; HCl salt, m. 200-1.degree. (PhCl) and some 10-(1'-diethylamino-2'-propyl)chlorophenothiazine. N-(.beta.-chloroethyl)piperidine (7.5 g.) in xylene (2 parts) added over 1 hr. to 11.6 g. I and 3 g. powd. NaOH in 60 cc. xylene at 125.degree.; processing as above gave 10-(2'-piperidinoethyl)chlorophenothiazine, b2.5, HCl salt, m. 187-8.degree. (PhCl).

L7 ANSWER 6 OF 29 CAPLUS COPYRIGHT 2003 ACS on STN

ACCESSION NUMBER: 1959:7170 CAPLUS  
DOCUMENT NUMBER: 53:7170  
ORIGINAL REFERENCE NO.: 53:1388b-c  
TITLE: Phenothiazine derivatives  
INVENTOR(S): **Charpentier, Paul**  
PATENT ASSIGNEE(S): Societe des usines chimiques Rhone-Poulenc  
DOCUMENT TYPE: Patent  
LANGUAGE: Unavailable  
FAMILY ACC. NUM. COUNT: 1  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
FR 1021280		19530217	FR	

AB N-(.beta.-Chloroethyl)phenothiazine (2.6 g.) and 0.9 g. Me<sub>2</sub>N(CH<sub>2</sub>)<sub>6</sub>NMe<sub>2</sub> heated 48 hrs. on a water bath, and the product taken up in 10 cc. EtOH and 10 cc. H<sub>2</sub>O gave after evapn. of the H<sub>2</sub>O from the aq. layer 1,6-hexamethylenebis {dimethyl[2-(10-phenothiazinyl)ethyl] diam-monium dichloride as a glue. The solid compd., m. 183.degree. (AcOEt), was

obtained by taking up the glue in ether. Cf. Fr. 942,366 (C.A. 45, 3427g).

L7 ANSWER 7 OF 29 CAPLUS COPYRIGHT 2003 ACS on STN

ACCESSION NUMBER: 1959:7167 CAPLUS  
DOCUMENT NUMBER: 53:7167  
ORIGINAL REFERENCE NO.: 53:1387g-i  
TITLE: Phenothiazines  
INVENTOR(S): **Charpentier, Paul**  
PATENT ASSIGNEE(S): Societe des usines chimiques Rhone-Poulenc  
DOCUMENT TYPE: Patent  
LANGUAGE: Unavailable  
FAMILY ACC. NUM. COUNT: 1  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
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FR 1075117		19541013	FR	
AB	10-(Dimethylaminoalkyl)chlorophenothiazines are prepd. by treating a haloalkylamine or its salt with chlorophenothiazine in the presence of a condensing agent; the chlorophenothiazine starting material is prepd. from m-chlorodiphenylamine and the position (1 or 3) of the Cl atom is not known. Thus, 7.5 g. Me <sub>2</sub> N(CH <sub>2</sub> ) <sub>3</sub> Cl in 7.5 g. xylene added over 1 hr. to 11.6 g. chlorophenothiazine and 2.4 g. NaNH <sub>2</sub> (80%) in 60 cc. boiling xylene, the mixt. refluxed 1 hr., cooled, estd. with dil. acid, the sq. exts. strongly basified, and the pptd. base extd. into Et <sub>2</sub> O and distd. gave 10-(3-dimethylaminopropyl)chlorophenothiazine, b <sub>0.8</sub> 200-5.degree.; HCl salt, m. 177-8.degree.. In the same way, the chlorophenothiazine with Me <sub>2</sub> NCH <sub>2</sub> CHClMe yields a mixt. of 10-(2-dimethylamino-1-propyl)chlorophenothiazine and 10-(1-dimethylamino-2-propyl)chlorophenothiazine contg. mainly the former; HCl salt, m. 236-7.degree. (abs. EtOH).			

L7 ANSWER 8 OF 29 CAPLUS COPYRIGHT 2003 ACS on STN

ACCESSION NUMBER: 1958:50854 CAPLUS  
DOCUMENT NUMBER: 52:50854  
ORIGINAL REFERENCE NO.: 52:9209h-i, 9210a  
TITLE: D-threo-1-(4-Nitrophenyl)-2-dichloroacetamido-1-propanol  
INVENTOR(S): **Charpentier, Paul**  
PATENT ASSIGNEE(S): Societe des usines chimiques Rhone-Poulenc  
DOCUMENT TYPE: Patent  
LANGUAGE: Unavailable  
FAMILY ACC. NUM. COUNT: 1  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
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FR 1041554		19531026	FR	
AB	The title compd. (I) is prepd. from 1-pseudonorephedrine (II) by direct or indirect nitration and then acylation with Cl <sub>2</sub> CHCO <sub>2</sub> Me. Thus, powd. II 1 is added slowly to a mixt. of H <sub>2</sub> SO <sub>4</sub> (66 B.acte.e.) 2.7 and HNO <sub>3</sub> (d. 1.4) 2.7 parts at 5.degree. yielding p-nitro-1-pseudonorephedrine (III), crystals from xylene, m. 149.degree., [ $\alpha$ ] <sub>D</sub> 19.6.degree. (3%, EtOH), after initial purification via the picrate, m. 165-6.degree.. III 1 and Cl <sub>2</sub> CHCO <sub>2</sub> Me 1.4 parts is heated 0.5 hr. on an H <sub>2</sub> O bath, the mixt. distd., and the residue crystd. from C <sub>6</sub> H <sub>6</sub> giving I, m. 97.degree., [ $\alpha$ ] <sub>D</sub> 44.degree. (3%, EtOH). Again II 1 in dry C <sub>5</sub> H <sub>5</sub> N 3 is refluxed with Ac <sub>2</sub> O 4 parts giving diacetyl-1-pseudonorephedrine, which 1 is dissolved in HNO <sub>3</sub> (d. 1.5) 4 parts at 0-5.degree., H <sub>2</sub> O added, the mixt. Et <sub>2</sub> O extd., the ext. evapd., and the residue refluxed 2 hrs. with 2N HCl 10 parts yielding HCl salt of III.			

L7 ANSWER 9 OF 29 CAPLUS COPYRIGHT 2003 ACS on STN  
 ACCESSION NUMBER: 1958:2161 CAPLUS  
 DOCUMENT NUMBER: 52:2161  
 ORIGINAL REFERENCE NO.: 52:446i,447a  
 TITLE: 9-(2-Diethylaminopropyl)thiaxanthene  
 INVENTOR(S): **Charpentier, Paul**  
 PATENT ASSIGNEE(S): Societe des usines chimiques Rhone-Poulenc  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Unavailable  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 815346		19511001	DE	

AB Thiaxanthene (I) is condensed with MeCHClCH<sub>2</sub>NEt<sub>2</sub> or MeCH(NEt<sub>2</sub>)CH<sub>2</sub>Cl in an aromatic hydrocarbon as solvent with a condensation agent which can form a metal deriv. with the labile H atom of I. The following compds. are suitable condensation agents: NaNH<sub>2</sub>, Me<sub>3</sub>CONa, LiBu, and PhLi, Thus, 25 g. I heated to boiling with 100 cc. xylene and 6,5 g. NaNH<sub>2</sub> (85%) under reflux, H<sub>2</sub>O and HCl added within 1 hr., the mixt. has cooled, the xylene layer sepd., the aq. layer neutralized with NaOH, and the pptd. base sepd. by ether extn., the ether evapd., and the residue rectified in vacuo gave 9-(2-diethylaminopropyl)thiaxanthene, b<sub>1</sub> 200.degree.. Cf. C.A. 39, 36302.

L7 ANSWER 10 OF 29 CAPLUS COPYRIGHT 2003 ACS on STN  
 ACCESSION NUMBER: 1957:47309 CAPLUS  
 DOCUMENT NUMBER: 51:47309  
 ORIGINAL REFERENCE NO.: 51:8811i,8812a-b  
 TITLE: Phenothiazine derivatives  
 INVENTOR(S): **Charpentier, Paul**  
 PATENT ASSIGNEE(S): Societe des usines chimiques Rhone-Poulenc  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Unavailable  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
FR 1003140		19520314	FR	

AB Phenothiazine derivs. are prepd. by treating the respective tertiary amine with an alkyl halide or an ester. Thus, 2.8 g. N-(2-di-methylamino-1-methylethyl)phenothiazine is dissolved in 5.6 g. C<sub>6</sub>H<sub>6</sub> and 2.8 g. MeI added, caused the mixt. to heat and an oily product to ppt.; the oil slowly crystd. to give 2-(10-phenothiazinyl)propyltrimethylammonium iodide, m. 206-7.degree.. In similar manner, the respective methosulfate, m. 149.degree., and the ethosulfate, m. 158.degree., were obtained. Also, 2-(10-phenothiazinyl)ethyltrimethylammonium iodide, m. 257.degree., the methosulfate, m. 155.degree., the chloride of the respective benzyldimethylammonium compd., m. 158.degree., the iodide of the methyldiethylammonium compd., m. 151.degree., the iodide of the triethylammonium compd., m. 227-8.degree., and 2-(10-phenothiazinyl)propyltrimethylammonium chloride, m. 267.degree., have been obtained. The compds. have antihistamine activity.

L7 ANSWER 11 OF 29 CAPLUS COPYRIGHT 2003 ACS on STN  
 ACCESSION NUMBER: 1956:40661 CAPLUS  
 DOCUMENT NUMBER: 50:40661  
 ORIGINAL REFERENCE NO.: 50:7880d-g  
 TITLE: (10-Dialkylaminoalkyl)phenothiazines  
 INVENTOR(S): **Charpentier, Paul**

PATENT ASSIGNEE(S): Societe des usines chimiques Rhone-Poulenc  
DOCUMENT TYPE: Patent  
LANGUAGE: Unavailable  
FAMILY ACC. NUM. COUNT: 1  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
FR 986718		19510803	FR	

AB In a process of prepg. 10-(dialkylaminoalkyl)phenothiazines, a dialkylaminoalkyl halide (free base) is treated with a concd. soln. of phenothiazine (I) in an aromatic hydrocarbon solvent, e.g. C<sub>6</sub>H<sub>6</sub>, PhMe, or C<sub>6</sub>H<sub>4</sub>Me<sub>2</sub> (II), in the presence of a condensing agent, e.g. NaNH<sub>2</sub>, metallic Na, powd. soda or potash, LiH, BuLi, PhLi, or LiCMe<sub>3</sub> (V), capable of forming a metalloorg. compd. with an org. compd. having a reactive H atom. Thus, 39.8 g. I and 5 g. Na in 160 cc. II refluxed 3 hrs., 26 g. Me<sub>2</sub>NCH<sub>2</sub>CH<sub>2</sub>Cl (III) mixed with an equal amt. of II added in portions within 1 hr. to the boiling soln., the soln. cooled, the product taken up in H<sub>2</sub>O, acidified with HCl, the II decanted, the aq. layer neutralized with soda and filtered, the filtrate made strongly alk., the pptd. base extd. by Et<sub>2</sub>O, the Et<sub>2</sub>O layer decanted, the solvent evapd., and the remainder distd. in vacuo gives 25 g. (46% calcd. on I) 10-(dimethylaminoethyl)phenothiazine (IV). Similarly, with powd. soda as condensing agent and PhMe as solvent, was obtained 86% (calcd. on I) IV. With Et<sub>2</sub>NCH<sub>2</sub>CH<sub>2</sub>Cl instead of III was obtained 77% 10-Et<sub>2</sub>NCH<sub>2</sub>CH<sub>2</sub> homolog of IV. Me<sub>2</sub>NCH<sub>2</sub>CHClMe instead of III gave 81% of a mixt. of the 10-Me<sub>2</sub>NCHMeCH<sub>2</sub> and 10-Me<sub>2</sub>NCH<sub>2</sub>CHMe analogs of IV; the 2 bases sepd. through the HCl salts gave 75% of the former one. Similar results are obtained by using anhyd. Et<sub>2</sub>O, which is then gradually replaced by II, as solvent, and PhLi as condensing agent, or by using V or LiH as the condensing agent in combination with II and PhMe, resp., as solvents. Cf. Fr. 917,595.

L7 ANSWER 12 OF 29 CAPLUS COPYRIGHT 2003 ACS on STN  
ACCESSION NUMBER: 1955:16278 CAPLUS  
DOCUMENT NUMBER: 49:16278  
ORIGINAL REFERENCE NO.: 49:3268h-i,3269a-i,3270a  
TITLE: 10-Dialkylaminoalkyl-2- or 4-chlorophenothiazines  
INVENTOR(S): Charpentier, Paul  
PATENT ASSIGNEE(S): Societe des usines chimiques Rhone-Poulenc  
DOCUMENT TYPE: Patent  
LANGUAGE: Unavailable  
FAMILY ACC. NUM. COUNT: 1  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2645640		19530714	US	

GI For diagram(s), see printed CA Issue.  
AB I, where R is H, Cl, Br, Me, or MeO in the 6- or 8-position, X is Cl or Br in the 2- or 4-position, A is a divalent, straight or branched aliphatic group contg. from 2 to 5 C atoms, and R<sub>1</sub> and R<sub>2</sub> are Me or Et or divalent groups which together form a pyrrolidine, piperidine or morpholine nucleus, are prepd. by the condensation of the corresponding phenothiazine with a dialkylaminoalkyl halide in the presence of NaNH<sub>2</sub> (II). The phenothiazines were prepd. by the cyclization in the presence of iodine of a substd. diphenylamine; e.g., m-ClC<sub>6</sub>H<sub>4</sub>-NHPh and S gave a mixt. (III) of 2- and 4-chlorophenothiazines. Thus, to a boiling suspension of 11.6 g. III [the 2 isomers m. at 196-8.degree. (IV), and 116-17.degree. (V), V in minor proportion, are not specifically identified], 2.4 g. 80% II, and 60 cc. xylene (VI) was added in 1 hr. 7.5 g. Me<sub>2</sub>N(CH<sub>2</sub>)<sub>3</sub>Cl (VII) in 7.5 g. VI, the whole refluxed 1 hr., cooled, treated with aq. HCl, the VI sepd., the HCl layer made strongly alk. with NaOH, the oil extd. with Et<sub>2</sub>O, and the

Et<sub>2</sub>O exts. concd. and distd. gave 10-(3-dimethylaminopropyl)-2- or 4-chlorophenothiazine (VIII), b<sub>0.8</sub> 200-5.degree.; hydrochloride (IX), m. 177-8.degree. (from PhCl); picrate (X), m. 172-3.degree.. The mother liquors from VIII give the isomer (XI) of VII; hydrochloride (XII), m. 169-70.degree.; picrate (XIII), m. 137-8.degree.. IV treated as above gave a base (XIV), b<sub>0.8</sub> 200-5.degree.; XIV gave IX and X. V treated as above except that finely powdered NaOH was used in place of II gave a base (XV), b<sub>0.7</sub> 203-9.degree.; XV gave XII. Employing II as in the first procedure above, the following 10-substituted 2- or 4-chlorophenothiazines were prepd. (side chain in 10-position, b.p. of base, m.p. of hydrochloride): mixt. of Me<sub>2</sub>NCHMeCH<sub>2</sub> and Me<sub>2</sub>NCH<sub>2</sub>CHMe, b<sub>0.9</sub> about 195.degree., 220-1.degree. (from alc.); Me<sub>2</sub>N(CH<sub>2</sub>)<sub>2</sub>, b<sub>2</sub> 215.degree., 165-6.degree. (from PhCl); mixt. of Et<sub>2</sub>NCHMeCH<sub>2</sub> and Et<sub>2</sub>NCH<sub>2</sub>CHMe, b<sub>3</sub> 230-5.degree., 200-1.degree. (from PhCl); Me<sub>2</sub>NCH<sub>2</sub>CMe<sub>2</sub>CH<sub>2</sub>, b<sub>3</sub> 220.degree., 182-3.degree. (from PhCl); Me<sub>2</sub>NCMe<sub>2</sub>(CH<sub>2</sub>)<sub>2</sub>, b<sub>0.1</sub> 195-205.degree. [base, m. 123 (from Et<sub>2</sub>O)]; Me<sub>2</sub>NCHEtCH<sub>2</sub>, b<sub>0.11</sub> 171-87.degree., 209-10.degree.; Me<sub>2</sub>NCHMe(CH<sub>2</sub>)<sub>2</sub>, b<sub>0.2</sub> 203-4.degree., 184-5.degree.; Me<sub>2</sub>NCMe<sub>2</sub>CH<sub>2</sub>, b<sub>0.1</sub> 170-85.degree., 240.degree.; Me<sub>2</sub>NCHEt(CH<sub>2</sub>)<sub>2</sub>, b<sub>0.15</sub> 203-6.degree., 131-3.degree. (oxalate); 2-pyrrolidinoethyl, b<sub>1.5</sub> 230.degree., 164.degree. (from PhCl); 2-morpholinoethyl, b<sub>2</sub> 260.degree., 192-3.degree. (from alc.); 3-pyrrolidinopropyl, b<sub>0.15</sub> 210.degree., -; (the following are 10-substituted bromophenothiazines; position of Br not stated) Me<sub>2</sub>N(CH<sub>2</sub>)<sub>3</sub>, b<sub>0.08</sub>, 186.degree., 170-1.degree.; mixt. of Me<sub>2</sub>NCHMeCH<sub>2</sub> and Me<sub>2</sub>NCH<sub>2</sub>CHMe, b<sub>0.15</sub> 190-2.degree., 203-4.degree. (from BuOH); [the following are 10-substituted dichlorophenothiazines; the dichlorophenothiazine, m. 259-60.degree., was prepd. from (3-ClC<sub>6</sub>H<sub>4</sub>)<sub>2</sub>NH, S and iodine at 180.degree.; LiH was used in place of II] Me<sub>2</sub>N(CH<sub>2</sub>)<sub>3</sub>, -, 215-18.degree. (from abs. alc.); Me<sub>2</sub>NCH<sub>2</sub>CMeCH<sub>2</sub>, base, m. 127-8.degree., 222-4.degree.; 3-pyrrolidinopropyl, b<sub>0.15</sub> 240.degree., 157-60.degree.; (the following is a 10-substituted methylchlorophenothiazine; the methylchlorophenothiazine, m. 267-70.degree., was prepd. from 3-MeC<sub>6</sub>H<sub>4</sub>NHC<sub>6</sub>H<sub>4</sub>Cl-3, S and iodine at 180.degree.; II was used) Me<sub>2</sub>N(CH<sub>2</sub>)<sub>3</sub>, b<sub>0.1</sub> 140-50.degree., 120.degree. (decompn.) (oxalate); (the following is a 10-substituted chloromethoxyphenothiazine; the chloromethoxyphenothiazine, m. 201-2.degree., was prepd. from 3-ClC<sub>6</sub>H<sub>4</sub>NHC<sub>6</sub>H<sub>4</sub>OMe-3, S and iodine at 140.degree.; LiH was used) 3-pyrrolidinopropyl, b<sub>0.1</sub> 208-10.degree., 172-3.degree. (oxalate) (from 50% aq. alc.). III and PhO(CH<sub>2</sub>)<sub>5</sub>Br in boiling VI in the presence of II gave 10-(5-phenoxy-1-pentyl)chlorophenothiazine (XVI), b<sub>1</sub> 255-60.degree.; XVI and AlCl<sub>3</sub> in CS<sub>2</sub> gave 10-(5-chloro-1-pentyl)chlorophenothiazine (XVII), b<sub>0.15</sub> 255.degree.; XVII (15 g.) and 20 g. anhyd. Me<sub>2</sub>NH mixed at 0.degree. in an autoclave, the whole agitated and kept 1 week at 20.degree., cooled in ice, the autoclave opened, the excess Me<sub>2</sub>NH evapd. the residual liquid taken up in 200 cc. H<sub>2</sub>O, the whole acidified to Congo red with 9 cc. HCl (d. 1.1), extd. at 80.degree. with 150 cc. PhMe, the acid layer sepd., made alk. with 11 cc. aq. Na<sub>2</sub>CO<sub>3</sub> (d. 1.33), the oil extd. with 100 cc. CHCl<sub>3</sub>, the exts. concd. and distd. gave 10-(5-dimethylamino-1-pentyl)chlorophenothiazine, b<sub>0.3</sub> 215-18.degree. (oxalate, m. 173-4.degree.). HO(CH<sub>2</sub>)<sub>4</sub>Cl, dihydropyran and several drops HCl gave 2-tetrahydropyranyl 4-chlorobutyl ether (XVIII), b<sub>2</sub> 90-3.degree.; XVIII, III, and II in VI gave as above 10-[4-(2-tetrahydropyranyloxy)-1-butyl]chlorophenothiazine (XIX), b<sub>0.3</sub> 230-48.degree.; XIX in 70% alc. with HCl gave 10-(4-hydroxy-1-butyl)chlorophenothiazine (XX); XX and SOCl<sub>2</sub> in C<sub>6</sub>H<sub>6</sub>, gave 10-(4-chloro-1-butyl)-chlorophenothiazine (XXI); and XXI and Me<sub>2</sub>NH as above gave 10-(4-dimethylamino-1-butyl)chlorophenothiazine, b<sub>0.16</sub> 210-14.degree. (hydrochloride, m. 161-2.degree.). The 10-lithio deriv. of chlorophenothiazine and Cl(CH<sub>2</sub>)<sub>3</sub>SO<sub>3</sub>SC<sub>6</sub>H<sub>4</sub>Me-p gave 10-[3 (p-toluene sulfonyloxy)propyl]chlorophenothiazine (XXII); subsequent hydrolysis, conversion of the 3-HO deriv. to the 3-chloro deriv. and reaction with Me<sub>2</sub>NH as above gave VIII, b<sub>0.8</sub> 200-10.degree., methylsulfomethylate, m. 140-1.degree.. These compds. are useful as pharmaceuticals and particularly as potentiators of other active pharmaceuticals.

L7 ANSWER 13 OF 29 CAPLUS COPYRIGHT 2003 ACS on STN

ACCESSION NUMBER: 1955:12377 CAPLUS  
DOCUMENT NUMBER: 49:12377  
ORIGINAL REFERENCE NO.: 49:2527a  
TITLE: 10-(Dialkylaminoalkyl)phenothiazines  
INVENTOR(S): **Charpentier, Paul**  
PATENT ASSIGNEE(S): Societe des usines chimiques Rhone-Poulenc  
DOCUMENT TYPE: Patent  
LANGUAGE: Unavailable  
FAMILY ACC. NUM. COUNT: 1  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
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DE 824944		19511217	DE	

AB See U.S. 2,519,886 (C.A. 45, 673c).

L7 ANSWER 14 OF 29 CAPLUS COPYRIGHT 2003 ACS on STN

ACCESSION NUMBER: 1953:41291 CAPLUS  
DOCUMENT NUMBER: 47:41291  
ORIGINAL REFERENCE NO.: 47:6949e-f  
TITLE: Substituted 10-(dimethylaminopropyl)phenothiazines  
AUTHOR(S): **Charpentier, Paul**; Gailliot, Paul; Jacob, Robert; Gaudechon, Jacques; Buisson, Paul  
SOURCE: Compt. rend. (1952), 235, 59-60  
DOCUMENT TYPE: Journal  
LANGUAGE: Unavailable

AB cf. C.A. 46, 7103e. The following derivs. of 10-(dimethylaminopropyl)phenothiazine are described: 1-Me (acid oxalate, m. 195-6.degree.; picrate, m. 141-2.degree.); 8-Me(HCl salt, m. 194.degree.; picrate, m. 143.degree.); 1-Cl(HCl salt, m. 169-70.degree.; picrate, m. 142-3.degree.); 3-Cl (HCl salt, m. 179-80.degree.; picrate, m. 171-2.degree.); 3-MeO (acid oxalate, m. 178-9.degree.; acid maleate, m. 140-1.degree.; picrate, m. 141-2.degree.). These derivs. have pronounced pharmacodynamic properties.

L7 ANSWER 15 OF 29 CAPLUS COPYRIGHT 2003 ACS on STN

ACCESSION NUMBER: 1953:6510 CAPLUS  
DOCUMENT NUMBER: 47:6510  
ORIGINAL REFERENCE NO.: 47:1194a-d  
TITLE: Phenothiazine derivatives  
INVENTOR(S): **Charpentier, Paul**  
PATENT ASSIGNEE(S): Societe des usines chimiques Rhone-Poulenc  
DOCUMENT TYPE: Patent  
LANGUAGE: Unavailable  
FAMILY ACC. NUM. COUNT: 1  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
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US 2595215		19520506	US	

AB New phenothiazine derivs. substituted in the 10-position with the group CH<sub>2</sub>CH(OH)CH<sub>2</sub>NR<sub>2</sub>, in which R is a lower alkyl group, are prepd. by condensing a secondary amine with 10-(2,3-epoxypropyl)phenothiazine (I) preferably at elevated temp. and pressure, with a solvent such as EtOH or MeOH. These new derivs. can be employed as spasmolytics, antihistaminics, vagolytics, local anesthetics, or as intermediates for other physiologically active substances. I is prepd. by adding 14 g. epichlorohydrin during 1.5 hrs. at 0.degree. to 20 g. phenothiazine in an Et<sub>2</sub>O soln. of LiPh (from 2 g. Li and 23 g. PhBr), letting stand overnight at room temp., adding H<sub>2</sub>O, drying the Et<sub>2</sub>O layer over Na<sub>2</sub>SO<sub>4</sub>, and distg.

off the Et<sub>2</sub>O; the residue is used for the reaction. Thus, I 12.7 and Me<sub>2</sub>NH 7 in 90% MeOH 15 g. are heated 12 hrs. at 120.degree. in a sealed tube, the excess Me<sub>2</sub>NH and MeOH removed on a H<sub>2</sub>O bath, the residue is taken up in dil. HCl, neutral products are removed by C<sub>6</sub>H<sub>6</sub> extn., and free 10-(3-dimethylamino-2-hydroxypropyl)phenothiazine obtained by addn. of NaOH and Et<sub>2</sub>O extn. Recrystn. from EtOAc-petr. ether gave the pure base, m. 84-5.degree.; HCl salt, m. 131.degree.. Also prepd. was 10-(3-diethylamino-2-hydroxypropyl)phenothiazine, m. 142-3.degree.; HCl salt, m. about 134.degree..

L7 ANSWER 16 OF 29 CAPLUS COPYRIGHT 2003 ACS on STN

ACCESSION NUMBER: 1952:42335 CAPLUS  
DOCUMENT NUMBER: 46:42335  
ORIGINAL REFERENCE NO.: 46:7103e-f  
TITLE: 10-(Diethylaminopropyl)phenothiazine isomers  
AUTHOR(S): **Charpentier, Paul**; Gailliot, Paul;  
Gaudechon, Jacques  
SOURCE: Compt. rend. (1951), 232, 2232-3  
DOCUMENT TYPE: Journal  
LANGUAGE: Unavailable

AB The structure of the 2 isomers, 10-(2-dimethylaminoisopropyl) (I) and 10-(2-dimethylaminopropyl)phenothiazine (II) and their resp. diethylamino homologs (Ia), (IIa), was detd. by degradation. I gave Me<sub>2</sub>NCH<sub>2</sub>CHBrMe (picrate, m. 113-14.degree.), and II gave MeCH(NMe<sub>2</sub>)CH<sub>2</sub>Br (picrate, m. 145-6.degree.); Ia and IIa gave the diethylamino analogs (picrates, m. 129-30.degree. and 114-15.degree., resp.).

L7 ANSWER 17 OF 29 CAPLUS COPYRIGHT 2003 ACS on STN

ACCESSION NUMBER: 1952:42334 CAPLUS  
DOCUMENT NUMBER: 46:42334  
ORIGINAL REFERENCE NO.: 46:7103d-e  
TITLE: Two isomeric 10-(dimethylaminopropyl)phenothiazines and their ethyl homologs  
AUTHOR(S): **Charpentier, Paul**; Ducrot, Rene  
SOURCE: Compt. rend. (1951), 232, 415-17  
DOCUMENT TYPE: Journal  
LANGUAGE: Unavailable

AB Correction abstr. to C.A. 45, 8536c. The last line should read "The antihistamine activity of II is from 3-10 times that of I."

L7 ANSWER 18 OF 29 CAPLUS COPYRIGHT 2003 ACS on STN

ACCESSION NUMBER: 1952:20642 CAPLUS  
DOCUMENT NUMBER: 46:20642  
ORIGINAL REFERENCE NO.: 46:3562d-f  
TITLE: Substituted barbituric acids and their preparation  
INVENTOR(S): **Charpentier, Paul**  
PATENT ASSIGNEE(S): Societe des usines chimiques Rhone-Poulenc  
DOCUMENT TYPE: Patent  
LANGUAGE: Unavailable  
FAMILY ACC. NUM. COUNT: 1  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
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US 2570024		19511002	US	

AB The prepn. of 5-m-bromophenyl-5-ethylbarbituric acid (I) and intermediates is covered. m-BrC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>Cl boiled in aq. EtOH with KCN yields m-BrC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>CN, b8 135-8.degree., converted by boiling with 96% EtOH in the presence of 66.degree. B.acte.e. H<sub>2</sub>SO<sub>4</sub> and neutralizing to Et (m-bromophenyl)acetate, b8 132-4.degree.. This (243 g.) and 153 g. (CO<sub>2</sub>Et)<sub>2</sub> added to 25.3 g. Na in 253 g. abs. EtOH yields after 24 hrs. the Na deriv. of the oxalyl deriv., which, treated with aq. H<sub>2</sub>SO<sub>4</sub> and extd.



with C<sub>6</sub>H<sub>6</sub>, yields after pyrolysis at 160-80.degree. di-Et (m-bromophenyl)malonate, b<sub>4</sub> 162-5.degree., which with EtONa, followed by EtI, yields the di-Et (m-bromophenyl)ethylmalonate, b<sub>2</sub> 157-9.degree.. This, refluxed with urea, MeOH, and Na with continuous distn. of the EtOH and MeOH, yields I, m. 252.degree. (from BuOH).

L7 ANSWER 19 OF 29 CAPLUS COPYRIGHT 2003 ACS on STN

ACCESSION NUMBER: 1951:49969 CAPLUS  
DOCUMENT NUMBER: 45:49969  
ORIGINAL REFERENCE NO.: 45:8536c-f  
TITLE: Two isomeric 10-(dimethylaminopropyl)phenothiazines and their ethyl homologs  
AUTHOR(S): **Charpentier, Paul**; Ducrot, Rene  
SOURCE: Compt. rend. (1951), 232, 415-17  
DOCUMENT TYPE: Journal  
LANGUAGE: Unavailable

AB cf. C.A. 42, 575e. 10-(2-Dimethylaminoisopropyl)phenothiazine (I)-HCl was obtained from the alc. mother liquor, from which 10-(2-dimethylaminopropyl)phenothiazine(II)-HCl had crystd. I was liberated from the I.HCl with soda and the picrate, crystd. from MeOH, brick-red, m. 166-7.degree., the same as the picrate of II. The pure bases were obtained from the picrates by treating with LiOH; I m. 64.degree. (from ether); I.HCl, m. 187-8.degree.. II is obtained in 2 cryst. forms: stable form (from, petr. ether), m. 70-1.degree.; unstable form (by evapn. of the ether soln. or by solidification), m. 60.degree., both forms gave the same picrate and also the same HCl salt, m. 203-5.degree.. Two analogous isomers, 10-(2-diethylamino-1-methylethyl)phenothiazine (III) and 10-(2-diethylaminopropyl)phenothiazine (IV) were obtained by the same method as I and II. IV sepd. readily, m. 55.degree., b<sub>0.5</sub> 190-3.degree. (yellow picrate, m. 151.degree.; HCl salt, m. 211.degree.); a small amt. of cryst. III was obtained (brick-red picrate, m. 145.degree.; HCl salt, m. 163.degree.). The toxicities of I and II to mice are of the same order, LD<sub>50</sub> 60-70 mg./kg. intravenously or 225-75 mg./kg. subcutaneously. The antihistamine activity of I is from 3-10 times that of II.

L7 ANSWER 20 OF 29 CAPLUS COPYRIGHT 2003 ACS on STN

ACCESSION NUMBER: 1951:41584 CAPLUS  
DOCUMENT NUMBER: 45:41584  
ORIGINAL REFERENCE NO.: 45:7144h-i  
TITLE: Sulfanilylthiourea  
INVENTOR(S): **Charpentier, Paul**  
PATENT ASSIGNEE(S): Societe des usines chimiques Rhone-Poulenc  
DOCUMENT TYPE: Patent  
LANGUAGE: Unavailable  
FAMILY ACC. NUM. COUNT: 1  
PATENT INFORMATION:

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
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	US 2545764		19510320	US	
AB	p-H <sub>2</sub> NC <sub>6</sub> H <sub>4</sub> SO <sub>2</sub> NHCN (I) and H <sub>2</sub> S are caused to react to give p-H <sub>2</sub> NC <sub>6</sub> H <sub>4</sub> SO <sub>2</sub> NHCSNH <sub>2</sub> (II). Thus, 5 g. I, 50 cc. abs. EtOH contg. 0.6 g. of NH <sub>3</sub> , and 0.2 g. "sulfurous chloride" are cooled to 0.degree., satd. with H <sub>2</sub> S, and heated 24 hrs. at 70.degree. to give II, m. about 250.degree. (decompn.). Similarly, 5 g. I, 50 cc. abs. EtOH contg. 2 g. of Et <sub>2</sub> NH, and 0.2 g. of tetramethylthiuram disulfide satd. at 0.degree. with H <sub>2</sub> S and heated 18 hrs. at 70.degree. give II.				

L7 ANSWER 21 OF 29 CAPLUS COPYRIGHT 2003 ACS on STN

ACCESSION NUMBER: 1951:19262 CAPLUS  
DOCUMENT NUMBER: 45:19262  
ORIGINAL REFERENCE NO.: 45:3428c-d

TITLE: 10-(2-Dimethylamino-1-propyl)phenothiazine  
 INVENTOR(S): **Charpentier, Paul**  
 PATENT ASSIGNEE(S): Societe des usines chimiques Rhone-Poulenc  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Unavailable  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2530451		19501121	US	

AB This compd., b3 190-2.degree., prepd. from phenothiazine, NaNH<sub>2</sub>, and Me<sub>2</sub>NCH<sub>2</sub>CHClMe or ClCH<sub>2</sub>CH(NMe<sub>2</sub>)Me in xylene, is useful as an antihistamine. Cf. C.A. 45, 673c; abstract from Compt. rend. in C.A. 42, 575e.

L7 ANSWER 22 OF 29 CAPLUS COPYRIGHT 2003 ACS on STN  
 ACCESSION NUMBER: 1951:19260 CAPLUS  
 DOCUMENT NUMBER: 45:19260  
 ORIGINAL REFERENCE NO.: 45:3427g-i,3428a  
 TITLE: Phenothiazine derivatives  
 INVENTOR(S): **Charpentier, Paul**  
 PATENT ASSIGNEE(S): Societe des usines chimiques Rhone-Poulenc  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Unavailable  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
FR 942366		19490207	FR	

GI For diagram(s), see printed CA Issue.  
 AB Compds. of pharmacodynamic interest have the general formula IA, where R<sub>1</sub> and R<sub>2</sub> are hydrocarbon residues, A and A' are bivalent aliphatic chains, and X is a halogen or acid residue. Ethylenebis(dimethyl[2-(10-phenothiazinyl)ethyl]ammonium bromide), m. 225-6.degree., is prepd. by heating 10-(2-dimethylaminoethyl)phenothiazine (I) 5.4 and C<sub>2</sub>H<sub>4</sub>Br<sub>2</sub> 1.7 g. for 6 hrs. on a water bath, extg. the unreacted reagents with C<sub>6</sub>H<sub>6</sub>, and crystg. from alc. I 5.4 in the same manner with Br(CH<sub>2</sub>)<sub>3</sub>Br 1, Br(CH<sub>2</sub>)<sub>4</sub>Br 1.9, Br(CH<sub>2</sub>)<sub>5</sub>Br 2, 1,4-dibromopentane 2, Br(CH<sub>2</sub>)<sub>6</sub>Br 2.2, Br(CH<sub>2</sub>)<sub>7</sub>Br 2.3 g. yields similar compds. in which A' = (CH<sub>2</sub>)<sub>3</sub>, m. 198-200.degree.; (CH<sub>2</sub>)<sub>4</sub>, m. 218.degree.; (CH<sub>2</sub>)<sub>5</sub>, m. 194-5.degree.; CHMe(CH<sub>2</sub>)<sub>3</sub>, m. 221.degree.; (CH<sub>2</sub>)<sub>6</sub>, m. 185-6.degree.; (CH<sub>2</sub>)<sub>7</sub>, m. 150.degree., resp.  
 10-(2-Dimethylamino-1-methylethyl)phenothiazine 5.7 with Br(CH<sub>2</sub>)<sub>5</sub>Br 2 and Br(CH<sub>2</sub>)<sub>6</sub>Br 2.2 g. yields, resp., pentamethylenebis(dimethyl[2-(10-phenothiazinyl)propyl]ammonium bromide), m. 279.degree., and a similar compd. in which A' = (CH<sub>2</sub>)<sub>6</sub>, m. 290.degree.. Pentamethylenebis(diethyl[2-(10-phenothiazinyl)ethyl]ammonium bromide), m. 140.degree. (from alc.-ether), is prepd. by heating 10-(2-diethylaminoethyl)phenothiazine 6 and Br(CH<sub>2</sub>)<sub>5</sub>Br 2.3 g. for 30 hrs. on a water bath, extg. with EtOAc, and evapg.

L7 ANSWER 23 OF 29 CAPLUS COPYRIGHT 2003 ACS on STN  
 ACCESSION NUMBER: 1951:13989 CAPLUS  
 DOCUMENT NUMBER: 45:13989  
 ORIGINAL REFERENCE NO.: 45:2511h-i  
 TITLE: Phenothiazines  
 INVENTOR(S): **Charpentier, Paul**  
 PATENT ASSIGNEE(S): Societe des usines chimiques Rhone-Poulenc  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Unavailable  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2526118		19501017	US	

AB Reflux phenothiazine 30, xylene 120, and 85% NaNH<sub>2</sub> 7, add gradually Et<sub>2</sub>NCH<sub>2</sub>CHClMe 28 in xylene 28 parts, boil 1 hr. longer, cool, dissolve in H<sub>2</sub>O 400 cc., acidify to Congo red with HCl, add NaOH to the aq. layer, ext. with Et<sub>2</sub>O, distil, collect the fraction b<sub>2</sub> 203-5.degree., convert the mixt. of the 2 isomers thus obtained in Me<sub>2</sub>CO or EtOAc with dry HCl into HCl salts, and sep. by crystn. 10-(2-Diethylaminopropyl)phenothiazine-HCl, m. 211.degree., crystallizes first; picrate, m. 148-9.degree.; methiodide, m. 238-9.degree.. From the mother liquid crystallizes 10-(2-diethylaminoisopropyl)phenothiazine-HCl, m. 162.degree.; picrate, m. 142-3.degree.; methiodide, m. 221.degree.. The first compd. is obtained exclusively if ClCH<sub>2</sub>CH(NEt<sub>2</sub>)Me is used. The substances are antihistaminics.

L7 ANSWER 24 OF 29 CAPLUS COPYRIGHT 2003 ACS on STN

ACCESSION NUMBER: 1951:3777 CAPLUS  
DOCUMENT NUMBER: 45:3777  
ORIGINAL REFERENCE NO.: 45:673c-e  
TITLE: 10-(Dialkylaminoalkyl)phenothiazines  
INVENTOR(S): **Charpentier, Paul**  
PATENT ASSIGNEE(S): Societe des usines chimiques Rhone-Poulenc  
DOCUMENT TYPE: Patent  
LANGUAGE: Unavailable  
FAMILY ACC. NUM. COUNT: 1  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2519886		19500822	US	

AB Phenothiazine 60, xylene 240, and NaNH<sub>2</sub> 14 g. refluxed, 40 g. Et<sub>2</sub>N(CH<sub>2</sub>)<sub>2</sub>Cl added in 1.5 hrs., and the mixt. refluxed 1 hr. give 63 g. 10-(2-diethylaminoethyl)phenothiazine (I), b<sub>1</sub> 1 200-5.degree. (HCl salt, m. 174-5.degree.). Similarly are obtained these 10-substituted analogs of I: 2-dimethylaminoethyl, b<sub>1</sub> 183-7.degree. (HCl salt, m. 201-1.5.degree.); 3-dimethylaminopropyl, b<sub>3</sub> 208-10.degree. (HCl salt, m. 181.degree.); 3-diethylaminopropyl, b<sub>1</sub> 5 213-15.degree.; 2-dimethylamino-1-methylethyl, b<sub>3</sub> 190-2.degree. (HCl salt, m. 204.degree.); 3-dimethylamino-2,2-dimethylpropyl, b<sub>3</sub> 196-9.degree.. 2-Methoxyphenothiazine similarly gives 10-(2-dimethylaminoethyl)-2-methoxyphenothiazine, b<sub>3</sub> 220-3.degree. (HCl salt m. 182.degree.), and the 10-(2-dimethylamino-1-methylethyl) analog, b<sub>3</sub> 218-22.degree.. These compds. are useful as antihistamine agents. Cf. C.A. 44, 1145h.

L7 ANSWER 25 OF 29 CAPLUS COPYRIGHT 2003 ACS on STN

ACCESSION NUMBER: 1950:25006 CAPLUS  
DOCUMENT NUMBER: 44:25006  
ORIGINAL REFERENCE NO.: 44:4928d-f  
TITLE: Benzene sulfonylthiourea  
INVENTOR(S): Arquet, Maurice; **Charpentier, Paul**  
PATENT ASSIGNEE(S): Soc. des usines chim. Rhone-Poulenc  
DOCUMENT TYPE: Patent  
LANGUAGE: Unavailable  
FAMILY ACC. NUM. COUNT: 1  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2498782		19500228	US	

AB The action of H<sub>2</sub>S on an arylsulfonylcyanamide yields a

phenylsulfonylthiourea. Thus p-H<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>SO<sub>2</sub>NHCN 19.7 g. in anhyd. C<sub>5</sub>H<sub>5</sub>N 160 g. is satd. at 0.degree. with H<sub>2</sub>S, heated in sealed tubes on the water bath several hours, cooled, the excess H<sub>2</sub>S and C<sub>5</sub>H<sub>5</sub>N removed in vacuo, and the residue taken up with water and acidified with AcOH to ppt. (aminophenylsulfonyl)thiourea, m. 200-1.degree.. Similarly prepd. were (phenylsulfonyl)thiourea, m. 138-9.degree., and the p-acetamido deriv., m. 228.degree.. Anhyd. NH<sub>3</sub> or NaOH soln. can be used in place of C<sub>5</sub>H<sub>5</sub>N. The products are therapeutically useful.

L7 ANSWER 26 OF 29 CAPLUS COPYRIGHT 2003 ACS on STN

ACCESSION NUMBER: 1950:5691 CAPLUS  
DOCUMENT NUMBER: 44:5691  
ORIGINAL REFERENCE NO.: 44:1145h-i  
TITLE: Alkylene dihalide salts of 10-(dialkylaminoalkyl)phenothiazines  
INVENTOR(S): Charpentier, Paul  
PATENT ASSIGNEE(S): Soc. des usines chim. Rhone-Poulenc  
DOCUMENT TYPE: Patent  
LANGUAGE: Unavailable  
FAMILY ACC. NUM. COUNT: 1  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2480355		19490830	US	

AB Therapeutically useful quaternary salts from dihaloalkanes of 2-7 C atoms and 10-(dialkylaminoalkyl)phenothiazines are prepd. by heating the reactants several hrs. at 100.degree., extg. with C<sub>6</sub>H<sub>6</sub> to remove the reactants, and recrystg. the products from EtOH. Thus are obtained these salts of 10-(2-dimethylaminoethyl)phenothiazine with Br(CH<sub>2</sub>)<sub>n</sub>Br, where n is: 2, m. 225-6.degree.; 4, m. 218.degree.; 5, m. 194-5.degree.; 6, m. 185-6.degree.; and 7, m. 150.degree.; and with Cl(CH<sub>2</sub>)<sub>3</sub>Cl, m. 198-200.degree., and MeCHBr(CH<sub>2</sub>)<sub>3</sub>Br, m. 221.degree.. The salt of 10-(2-diethylaminoethyl)phenothiazine with Br(CH<sub>2</sub>)<sub>5</sub>Br m. 140.degree., and of 10-(2-dimethylaminoisopropyl)phenothiazine with Br-(CH<sub>2</sub>)<sub>5</sub>Br m. 279.degree., and with Br-(CH<sub>2</sub>)<sub>6</sub>Br m. 290.degree.. Cf. C.A. 42, 575e.

L7 ANSWER 27 OF 29 CAPLUS COPYRIGHT 2003 ACS on STN

ACCESSION NUMBER: 1948:2627 CAPLUS  
DOCUMENT NUMBER: 42:2627  
ORIGINAL REFERENCE NO.: 42:575e-f  
TITLE: The constitution of a 10-(dimethylaminopropyl)phenothiazine  
AUTHOR(S): Charpentier, Paul  
SOURCE: Compt. rend. (1947), 225, 306-8  
DOCUMENT TYPE: Journal  
LANGUAGE: Unavailable

AB The compd. first reported (C.A. 41, 4234c) as 10-(2-dimethylaminoisopropyl)phenothiazine has now been shown to be the isomeric 10-(2-dimethylamino-1-propyl)phenothiazine (I). When subjected to exhaustive methylation it gave 10-propenylphenothiazine, b1 181-4.degree., identified by: (1) KMnO<sub>4</sub> oxidation in cold acetone to 10-formylphenothiazine, m. 144-5.degree., and (2) hydrolysis by boiling N H<sub>2</sub>SO<sub>4</sub> to give phenothiazine and EtCHO, thus proving the structure I for the compd. I m. 60.degree., b3 190-2.degree. (HCl salt, m. 203-4.degree.; methiodide, m. 206-7.degree.; methyl benzenesulfonate, m. 275.degree.). I was also prepd. from phenothiazine and ClCH<sub>2</sub>CHMeNMe<sub>2</sub>.

L7 ANSWER 28 OF 29 CAPLUS COPYRIGHT 2003 ACS on STN

ACCESSION NUMBER: 1935:13901 CAPLUS  
DOCUMENT NUMBER: 29:13901  
ORIGINAL REFERENCE NO.: 29:1771g-i

TITLE: The chlorobutene, C<sub>4</sub>H<sub>7</sub>Cl, which results from the action of phosphorus pentachloride on 2-butanone

AUTHOR(S): **Charpentier, Paul**

SOURCE: Bull. soc. chim. [5] (1934), 1, 1407-11

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

AB As early as 1875 (Ber. 8, 412) it was found that PCl<sub>5</sub> acted on MeCOCH<sub>2</sub>Me to give MeCCl<sub>2</sub>CH<sub>2</sub>Me, which partially lost HCl to give C<sub>4</sub>H<sub>7</sub>Cl. Since the structure of the latter was in doubt C. repeated the expt. and obtained 60% 2,2-dichlorobutane, b. 102.degree., d. 1.05 and 40% of a liquid, b. 60-5.degree.. This proved to be a mixt. which could not be completely sepd. by distn. After repeated distns. the 2 end fractions were oxidized with KMnO<sub>4</sub> and one (b. 58.5-9.degree.) gave mainly EtCO<sub>2</sub>H and the other (65-6.degree.) mainly AcOH. Hence the MeCCl<sub>2</sub>CH<sub>2</sub>Me lost HCl to form both MeCCl:CHMe and H<sub>2</sub>C:CClCH<sub>2</sub>Me but the relative amts. could not be detd.

L7 ANSWER 29 OF 29 CAPLUS COPYRIGHT 2003 ACS on STN

ACCESSION NUMBER: 1934:46083 CAPLUS

DOCUMENT NUMBER: 28:46083

ORIGINAL REFERENCE NO.: 28:5598d-e

TITLE: The system neonal and pyramidone

AUTHOR(S): **Charpentier, Paul**

SOURCE: Bulletin des Sciences Pharmacologiques (1934), 41, 328-30

CODEN: BSPHAV; ISSN: 0366-3493

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

AB Both substances can be detd. by acidimetric titration: Neonal is monoacid to thymolphthalein and neutral to methyl orange; pyramidone is monobasic to methyl orange and neutral to thymolphthalein. The mixt. can be prepd. in benzine, EtOH or H<sub>2</sub>O. The curve of the m. ps. of mixts. shows a max. at an equimol. compn. at 108-110.degree..

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COST IN U.S. DOLLARS	SINCE FILE ENTRY	TOTAL SESSION
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=> s (supercritical or critical or fluid) (4a) carbon dioxide  
L8 17437 (SUPERCRITICAL OR CRITICAL OR FLUID) (4A) CARBON DIOXIDE

=> s (continuous or continual?) (3a) (polymerization or polymerizing)  
L9 11165 (CONTINUOUS OR CONTINUAL?) (3A) (POLYMERIZATION OR POLYMERIZING)

=> s 18 and 19

L10 41 L8 AND L9

=> s 110 and recycl? or return?

L11 1154144 L10 AND RECYCL? OR RETURN?

=> s 110 and (recycl? or return?) (8a) (reactor or vessel)

L12 8 L10 AND (RECYCL? OR RETURN?) (8A) (REACTOR OR VESSEL)

=> d 112 1-8 ibib abs

L12 ANSWER 1 OF 8 USPATFULL on STN

ACCESSION NUMBER: 2003:105780 USPATFULL

TITLE: Continuous method and apparatus for separating polymer from a high pressure **carbon dioxide** fluid stream

INVENTOR(S): Royer, Joseph R., Greenville, SC, UNITED STATES  
Roberts, George W., Raleigh, NC, UNITED STATES

	NUMBER	KIND	DATE
PATENT INFORMATION:	US 2003072690	A1	20030417
APPLICATION INFO.:	US 2002-82373	A1	20020225 (10)
RELATED APPLN. INFO.:	Continuation-in-part of Ser. No. US 2000-709206, filed on 9 Nov 2000, PENDING		

	NUMBER	DATE
PRIORITY INFORMATION:	US 1999-165177P	19991112 (60)
DOCUMENT TYPE:	Utility	
FILE SEGMENT:	APPLICATION	
LEGAL REPRESENTATIVE:	MYERS BIGEL SIBLEY & SAJOVEC, PO BOX 37428, RALEIGH, NC, 27627	
NUMBER OF CLAIMS:	34	
EXEMPLARY CLAIM:	1	
NUMBER OF DRAWINGS:	15 Drawing Page(s)	
LINE COUNT:	1487	

CAS INDEXING IS AVAILABLE FOR THIS PATENT.

AB A method for continuously separating polymer from a high pressure fluid stream comprises subjecting the high pressure fluid stream comprising polymer particles to a filter, wherein the filter segregates the high pressure fluid stream from the polymer particles; subjecting the polymer particles to a rotating device which transports the polymer particles away from the filter, wherein the polymer particles are exposed to thermal conditions sufficient to melt the polymer particles and form a seal surrounding at least a portion of the rotating device; and separating the molten polymer from the rotating device. The method is carried out such that the separation of polymer from the high pressure fluid stream occurs under steady-state.

CAS INDEXING IS AVAILABLE FOR THIS PATENT.

L12 ANSWER 2 OF 8 USPATFULL on STN

ACCESSION NUMBER: 2002:137113 USPATFULL

TITLE: Process for heterophase reactions in a liquid or supercritical dispersion medium

INVENTOR(S): Hergeth, Wolf-Dieter, Julbach, GERMANY, FEDERAL REPUBLIC OF  
Becker, Richard, Emmerting, GERMANY, FEDERAL REPUBLIC OF

Gunaltay, Mehmet, Emmerting, GERMANY, FEDERAL REPUBLIC  
 OF  
 Jekat, Herbert, Unterwoessen, GERMANY, FEDERAL REPUBLIC  
 OF  
 Rindfleisch, Frank, Koeln-Fuehlingen, GERMANY, FEDERAL  
 REPUBLIC OF  
 Wendenburg, Heribert, Winhoering, GERMANY, FEDERAL  
 REPUBLIC OF  
 PATENT ASSIGNEE(S): Wacker Chemie GmbH, Munich, GERMANY, FEDERAL REPUBLIC  
 OF (non-U.S. corporation)

	NUMBER	KIND	DATE
PATENT INFORMATION:	US 6403729	B1	20020611
APPLICATION INFO.:	US 1999-417618		19991013 (9)

	NUMBER	DATE
PRIORITY INFORMATION:	DE 1998-19847822	19981016
DOCUMENT TYPE:	Utility	
FILE SEGMENT:	GRANTED	
PRIMARY EXAMINER:	Wu, David W.	
ASSISTANT EXAMINER:	Cheung, William K.	
LEGAL REPRESENTATIVE:	Brooks & Kushman P.C.	
NUMBER OF CLAIMS:	3	
EXEMPLARY CLAIM:	1	
NUMBER OF DRAWINGS:	4 Drawing Figure(s); 3 Drawing Page(s)	
LINE COUNT:	484	

CAS INDEXING IS AVAILABLE FOR THIS PATENT.

AB The invention provides a process for heterophase reactions in a liquid  
 or supercritical dispersion medium, in which the starting materials are  
 introduced into the reaction zone of a reactor provided with one or more  
 enrichment zones and the reaction product or unreacted starting material  
 or both starting material and reaction product are discharged via one  
 enrichment zone in each case, wherein a) the reaction product together  
 with the dispersion medium is discharged from the reactor via an  
 enrichment zone, the reaction product is separated off and the  
 dispersion medium is, if desired in countercurrent to the reaction  
 product/dispersion medium mixture and via an enrichment zone, returned  
 to the reaction zone, or b) unreacted starting material together with  
 the dispersion medium is discharged, the starting material is separated  
 from the dispersion medium and returned directly to the reaction zone  
 and the dispersion medium is, if desired, in countercurrent to the  
 reaction product/dispersion medium mixture and via an enrichment zone,  
 returned to the reaction zone, or the steps a) and b) are combined with  
 one another.

CAS INDEXING IS AVAILABLE FOR THIS PATENT.

L12 ANSWER 3 OF 8 USPATFULL on STN

ACCESSION NUMBER: 2002:72978 USPATFULL

TITLE: Multimodal fluoropolymers and methods of making the same

INVENTOR(S): DeSimone, Joseph M., Chapel Hill, NC, UNITED STATES  
 Roberts, George W., Raleigh, NC, UNITED STATES  
 Charpentier, Paul A., London, CANADA

	NUMBER	KIND	DATE
PATENT INFORMATION:	US 2002040118	A1	20020404
APPLICATION INFO.:	US 2001-860413	A1	20010518 (9)

	NUMBER	DATE
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PRIORITY INFORMATION:	US 2000-205817P	20000519 (60)
DOCUMENT TYPE:	Utility	
FILE SEGMENT:	APPLICATION	
LEGAL REPRESENTATIVE:	MYERS BIGEL SIBLEY & SAJOVEC, PO BOX 37428, RALEIGH, NC, 27627	
NUMBER OF CLAIMS:	38	
EXEMPLARY CLAIM:	1	
NUMBER OF DRAWINGS:	7 Drawing Page(s)	
LINE COUNT:	668	

CAS INDEXING IS AVAILABLE FOR THIS PATENT.

AB A method for forming a fluoropolymer comprises providing a reaction mixture comprising carbon dioxide, at least one fluoromonomer, and an initiator; and reacting the at least one fluoromonomer in the reaction mixture to form a fluoropolymer. The fluoropolymer has a multimodal molecular weight distribution.

CAS INDEXING IS AVAILABLE FOR THIS PATENT.

L12 ANSWER 4 OF 8 USPATFULL on STN

ACCESSION NUMBER:	2000:131965	USPATFULL
TITLE:	Pressure polymerization of polyester	
INVENTOR(S):	Maurer, Charles J., Matthews, NC, United States Shaw, Gordon, Charlotte, NC, United States Smith, Vicky S., Greenville, SC, United States	
PATENT ASSIGNEE(S):	Arteva North America S.A.R.L., Zurich, Switzerland (non-U.S. corporation)	

	NUMBER	KIND	DATE
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PATENT INFORMATION:	US 6127493		20001003
APPLICATION INFO.:	US 1998-185998		19981104 (9)
DOCUMENT TYPE:	Utility		
FILE SEGMENT:	Granted		
PRIMARY EXAMINER:	Acquah, Samuel A.		
LEGAL REPRESENTATIVE:	Clements, Gregory N.		
NUMBER OF CLAIMS:	44		
EXEMPLARY CLAIM:	1		
NUMBER OF DRAWINGS:	2 Drawing Figure(s); 2 Drawing Page(s)		
LINE COUNT:	1089		

CAS INDEXING IS AVAILABLE FOR THIS PATENT.

AB A process is disclosed for the preparation of a polyester polymer or polyester copolymer under superatmospheric pressure conditions in a pipe or tubular reaction under turbannular flow conditions. Reaction material having a glycol equivalents to carboxylic acid equivalents mole ratio of from 1.0:1 to 1.2:1, together with a superatmospheric dense gaseous medium are fed co-currently to the reactor. Dicarboxylic acid and/or diol raw materials may be injected into any of the reaction zones in the process during operation to achieve the overall desired mole ratio balance. The process operates at temperatures of from about 220.degree. C. to about 320.degree. C., with turbannular flow achieved before the polymer product and gas exit the reactor process. The pressure in the reaction zones can be in the range from 15 psia to 2500 psia. A polymer product having a DP of a greater than 40, more preferably at least about 70, is achieved by the transfer of water from the reacting material polymer melt to the gaseous medium in the reactor.

CAS INDEXING IS AVAILABLE FOR THIS PATENT.

L12 ANSWER 5 OF 8 USPATFULL on STN

ACCESSION NUMBER:	2000:114090	USPATFULL
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TITLE: Pressure polymerization of polyester  
 INVENTOR(S): Maurer, Charles J., Matthews, NC, United States  
 Shaw, Gordon, Charlotte, NC, United States  
 Smith, Vicky S., Greer, SC, United States  
 Buelow, Steven J., Los Alamos, NM, United States  
 Tumas, William, Los Alamos, NM, United States  
 Contreras, Veronica, San Antonio, TX, United States  
 Martinez, Ronald J., Santa Cruz, NM, United States  
 PATENT ASSIGNEE(S): The Regents of the University of California, Los  
 Alamos, NM, United States (U.S. corporation)

	NUMBER	KIND	DATE
PATENT INFORMATION:	US 6111064		20000829
APPLICATION INFO.:	US 1999-325442		19990603 (9)
RELATED APPLN. INFO.:	Continuation of Ser. No. US 1998-185998, filed on 4 Nov 1998		
DOCUMENT TYPE:	Utility		
FILE SEGMENT:	Granted		
PRIMARY EXAMINER:	Acquah, Samuel A.		
LEGAL REPRESENTATIVE:	Cottrell, Bruce H.		
NUMBER OF CLAIMS:	2		
EXEMPLARY CLAIM:	1		
NUMBER OF DRAWINGS:	2 Drawing Figure(s); 2 Drawing Page(s)		
LINE COUNT:	894		

CAS INDEXING IS AVAILABLE FOR THIS PATENT.

AB A process is disclosed for the preparation of a polyester polymer or polyester copolymer under superatmospheric pressure conditions in a pipe or tubular reaction under turbannular flow conditions. Reaction material having a glycol equivalents to carboxylic acid equivalents mole ratio of from 1.0:1 to 1.2:1, together with a superatmospheric dense gaseous medium are fed co-currently to the reactor. Dicarboxylic acid and/or diol raw materials may be injected into any of the reaction zones in the process during operation to achieve the overall desired mole ratio balance. The process operates at temperatures of from about 220.degree. C. to about 320.degree. C., with turbannular flow achieved before the polymer product and gas exit the reactor process. The pressure in the reaction zones can be in the range from 15 psia to 2500 psia. A polymer product having a DP of a greater than 40, more preferably at least about 70, is achieved by the transfer of water from the reacting material polymer melt to the gaseous medium in the reactor.

CAS INDEXING IS AVAILABLE FOR THIS PATENT.

L12 ANSWER 6 OF 8 USPATFULL on STN  
 ACCESSION NUMBER: 95:18539 USPATFULL  
 TITLE: Production of polyesters and polyester articles having good clarity  
 INVENTOR(S): Yau, Cheuk C., Kingsport, TN, United States  
 Cherry, Clinton, Kingsport, TN, United States  
 PATENT ASSIGNEE(S): Eastman Chemical Company, Kingsport, TN, United States  
 (U.S. corporation)

	NUMBER	KIND	DATE
PATENT INFORMATION:	US 5393871		19950228
APPLICATION INFO.:	US 1993-316		19930104 (8)
DOCUMENT TYPE:	Utility		
FILE SEGMENT:	Granted		
PRIMARY EXAMINER:	Acquah, Samuel A.		
LEGAL REPRESENTATIVE:	Boshears, Betty J., Gwinnell, Harry J.		
NUMBER OF CLAIMS:	14		

EXEMPLARY CLAIM: 1  
LINE COUNT: 391

CAS INDEXING IS AVAILABLE FOR THIS PATENT.

AB Disclosed is an improvement in a process for producing linear polyesters wherein a precursor polyester is first formed and subsequently the precursor polyester is formed into particles and further polymerized in the solid state, wherein the improvement comprises contacting the particles while at a temperature of about 140.degree. to about 2.degree. C. below the melting point of the polyester with the vapor of water or an organic compound having at least one OH group.

CAS INDEXING IS AVAILABLE FOR THIS PATENT.

L12 ANSWER 7 OF 8 USPATFULL on STN

ACCESSION NUMBER: 95:18531 USPATFULL  
TITLE: Production of branched polyesters  
INVENTOR(S): Yau, Cheuk C., Kingsport, TN, United States  
Cherry, Clinton, Kingsport, TN, United States  
PATENT ASSIGNEE(S): Eastman ChemicalI Company, Kingsport, TN, United States  
(U.S. corporation)

	NUMBER	KIND	DATE
PATENT INFORMATION:	US 5393863		19950228
APPLICATION INFO.:	US 1993-315		19930104 (8)
DOCUMENT TYPE:	Utility		
FILE SEGMENT:	Granted		
PRIMARY EXAMINER:	Acquah, Samuel A.		
LEGAL REPRESENTATIVE:	Boshears, Betty J., Gwinnell, Harry J.		
NUMBER OF CLAIMS:	14		
EXEMPLARY CLAIM:	1		
LINE COUNT:	406		

CAS INDEXING IS AVAILABLE FOR THIS PATENT.

AB Disclosed is an improvement in a process for producing branched polyesters wherein a precursor polyester is first formed and subsequently the precursor polyester is formed into particles and further polymerized in the solid state, wherein the improvement comprises contacting the particles while at a temperature of about 140.degree. to about 2.degree. C. below the melting point of the polyester with the vapor of water or an organic compound having at least one OH group.

CAS INDEXING IS AVAILABLE FOR THIS PATENT.

L12 ANSWER 8 OF 8 CAPLUS COPYRIGHT 2003 ACS on STN

ACCESSION NUMBER: 2001:360058 CAPLUS  
DOCUMENT NUMBER: 134:367383  
TITLE: Continuous process for making polymers in carbon dioxide  
INVENTOR(S): Charpentier, Paul A.; Desimone, Joseph M.; Roberts, George W.  
PATENT ASSIGNEE(S): North Carolina State University, USA; University of North Carolina at Chapel Hill  
SOURCE: PCT Int. Appl., 58 pp.  
CODEN: PIXXD2  
DOCUMENT TYPE: Patent  
LANGUAGE: English  
FAMILY ACC. NUM. COUNT: 2  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
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WO 2001034667 A1 20010517 WO 2000-US30765 20001110  
W: DE, ES, GB, IL, JP, LU, NO, PL, RO, RU, ZA  
RW: AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL,  
PT, SE, TR  
EP 1248807 A1 20021016 EP 2000-978446 20001110  
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT,  
IE, FI, RO, CY, TR  
JP 2003514083 T2 20030415 JP 2001-537378 20001110  
PRIORITY APPLN. INFO.: US 1999-165177P P 19991112  
WO 2000-US30765 W 20001110

AB A method for carrying out the **continuous polymn.** of a monomer in a carbon dioxide reaction medium comprises the steps of: (a) providing an app. including a continuous reaction vessel and a separator; (b) carrying out a polymn. reaction in the reaction vessel by combining a monomer and a carbon dioxide reaction medium therein (and preferably by also combining an initiator therein), wherein the reaction medium is a liq. or supercrit. fluid, and wherein the reaction produces a solid polymer product in the reaction vessel; then (c) withdrawing a continuous effluent stream from the reaction vessel during the polymn. reaction, wherein the effluent stream is maintained as a liq. or supercrit. fluid; then (d) passing the continuous effluent stream through the separator and sepg. the solid polymer therefrom while maintaining at least a portion of the effluent stream as a liq. or supercrit. fluid; and then (e) returning at least a portion of the continuous effluent stream to the reaction vessel while maintaining the effluent stream as a liq. or supercrit. fluid. The need for significant recompression of the continuous effluent stream prior to **return** to the reaction vessel is thereby minimized. App. for carrying out such methods is also disclosed.

REFERENCE COUNT: 6 THERE ARE 6 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

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L12 ANSWER 2 OF 8 USPATFULL on STN

SUMM A frequently employed solvent and/or dispersion medium is **carbon dioxide** in the liquid or **supercritical** state (D.A. Canelas, J. M. DeSimone: Adv. Polym. Sci. 133 (1997) 103). The **supercritical** state of **carbon dioxide** can be achieved without an excessively high engineering effort. The values for the critical point are T.sub.crit(CO.sub.2)=31.1.degree. C. and p.sub.crit(CO.sub.2)=73.8 bar. The **supercritical** state of **carbon dioxide** is characterized by a very low viscosity and a readily variable density, by means of which the solvent properties of the carbon dioxide can also be varied. Reviews of polymerization processes in liquid and **supercritical carbon dioxide** may be found in A. I. Cooper, J. M. DeSimone: Curr. Opin. Solid State Mater. Sci. 1 (1996) 6. The separation of the polymer from the supercritical fluid can be carried out by means of separation processes which are known in principle (e.g. EP-B 159021).

SUMM It is an object of the present invention to provide a process and an apparatus for heterophase reactions, in particular, for dispersion polymerizations in a liquid or **supercritical carbon dioxide**, which offers the possibility of carrying out the reactions continuously, semi-continuously or batchwise and the possibility of concentrating the reaction product and re-using unreacted starting materials.

DRWD FIG. 2 is a flow diagram of the process for **continuous dispersion polymerization** in carbon dioxide.

DETD The process is suitable for carrying out any heterophase reaction.

Preference is given to precipitation reactions, crystallizations and, particularly preferably, heterophase polymerizations. Greatest preference is given to the dispersion -polymerization of ethylenically unsaturated monomers in liquid or **supercritical carbon dioxide**.

DETD The most preferred dispersion polymerization can be carried out either in the liquid state of the dispersion medium **carbon dioxide** or in the **supercritical** state of the dispersion medium carbon dioxide. The values for the triple point of carbon dioxide are  $T_{\text{sub.trip}}(\text{CO}_{\text{sub.2}}) = -56.6^{\circ}\text{C}$ . and  $p_{\text{sub.trip}}(\text{CO}_{\text{sub.2}}) = 5.2$  bar. The liquid state can be achieved at pressures greater than 5.2 bar and temperatures below the phase boundary curve between the triple point and the critical point. The values for the critical point are  $T_{\text{sub.crit}}(\text{CO}_{\text{sub.2}}) = -31.1^{\circ}\text{C}$ . and  $p_{\text{sub.crit}}(\text{CO}_{\text{sub.2}}) = 73.8$  bar.

DETD At pressures and temperatures greater than the **critical** values, the **carbon dioxide** is present in a **supercritical** state. The polymerizations are preferably carried out in the pressure range from 74 to 600 bar. The preferred polymerization temperatures are in the range from  $31^{\circ}\text{C}$ . to  $200^{\circ}\text{C}$ . The carbon dioxide is used in an amount of from 0.5 to 2000 parts by weight of  $\text{CO}_{\text{sub.2}}$ , preferably from 10 to 600 parts by weight of  $\text{CO}_{\text{sub.2}}$ , in each case based on the total weight of the monomers.

DETD In the preferred embodiment, an enrichment zone for unreacted starting material is located at the end of the reaction zone opposite to the enrichment zone for the reaction product. The unreacted starting material dispersed in the dispersion medium is discharged via the enrichment zone and is separated stepwise from the dispersion medium in the individual separation stages. All or some of the unreacted starting material is **returned** to the **reactor**. The dispersion medium is circulated in the described way, namely by introduction into the enrichment zone for reaction product.

DETD FIG. 2 shows a flow diagram of the plant according to the invention for **continuous dispersion polymerization** in carbon dioxide.

DETD The carbon dioxide which has been de-pressurized to gas pressure is conveyed from the de-pressurization and collection vessel (55) via a filter (66) to separate off residual product and a line (67) to the de-pressurization and separation **vessel** (38) and thus **returned** to the carbon dioxide circuit.

CLM What is claimed is:

2. The process as claimed in claim 1, wherein a) the reaction product together with the dispersion medium is discharged from the reactor via an enrichment zone, the reaction product -is separated off and the dispersion medium is **returned** to the **reactor** in countercurrent to the starting material/dispersion medium mixture, and b) unreacted starting material is discharged together with the dispersion medium, the starting material is separated from the dispersion medium and returned directly to the reaction zone and the dispersion medium is returned to the reaction zone via an enrichment zone.

3. The process as claimed in claim 1, wherein the ethylenically unsaturated monomers are polymerized in liquid or **supercritical carbon dioxide** by means of a dispersion polymerization.

L12 ANSWER 4 OF 8 USPATFULL on STN

SUMM WO 96/39456 to DeSimone discloses a transesterification-polycondensation process for polyester using carbon dioxide as the polymerization medium. The polycondensation of BHET conducted in an autoclave with flowing **supercritical carbon dioxide** is exemplified.

Ethylene glycol is liberated for every step growth in DP. A 10-50 mole percent excess of glycol is recommended. The highest DP achieved in the examples is 33. As exemplified by DeSimone, excess diol is removed by flowing carbon dioxide or by including a surfactant with carbon dioxide capable of scavenging the condensate within the reactor without removing carbon dioxide. Higher DP increases would be desirable without requiring purification of the diol to remove the surfactant. Furthermore, as demonstrated by DeSimone, a DP of about 33 was achieved in a continuous flow process starting with BHET. However, this required a relatively large amount of carbon dioxide, i.e., a weight ratio of carbon dioxide to BHET of 43:1.

SUMM In accordance with another aspect of the invention, there is provided in two or more stages using elongated tubular reaction zones, a **continuous** process for **polymerization** of polyester precursors in the presence of a gaseous compound which has a low temperature atmospheric boiling point (<100.degree. C.). Reaction material is fed to the first zone, which is operated at from 220.degree. C. to 320.degree. C., having a number average DP of 1 and comprising 50-100 mole percent mono(hydroxyalkyl) ester of a dicarboxylic acid and 0-50 mole percent of bis(hydroxyalkyl) ester of a dicarboxylic acid. In any subsequent zone the reaction product of the previous zone is further reacted. Preferably the ratio of the cross-sectional area of the zones divided by the wetted perimeter is greater than 2.5 cm.

DRWD FIG. 2 is a schematic diagram of a **continuous** **polymerization** process according to the invention using first stage and second stage tubular reactors.

DETD With reference to FIG. 2 wherein like numerals depict like structures, a **continuous polymerization** process is provided which is not a continuous pipe reactor, but a staged tubular reactor. The materials of construction for the vessels are beyond the scope of the disclosure, but stainless steel 304 and 316, nickel alloys including types C-276, 265, 825 and 620, and ceramic coated metals are suitable. Reaction material is fed through conduit 1 to vessel 20. Compressed gaseous compound is also fed to vessel 20 through conduit 2. A dense gas/reaction material slurry is fed through conduit 3 to tubular reactor 21 where reaction occurs and the water of reaction is transferred into the gas phase, increasing the degree of polymerization of the polymeric material. From reactor 21, the two phase material enters separation vessel 22 where the dense gas is separated from the polymer by conventional means such as by gravimetric separation. The polymer leaves the vessel from the bottom through conduit 5, and the gas flows out of the top of vessel 22 through a pressure regulating device, for example a control valve. The de-pressurized gas is fed via conduit 8 to clean-up unit 25, which may be composed of one or more vessels, where water and other compounds are removed from the gas by conventional means. Any reaction materials removed are discharged from the base of vessel 25 via conduit 10 for recovery, and any by-product gases via other conduits, not shown. The clean gas is then fed through conduit 9 to compressor 26. Clean pressurized gas enters the second stage tubular reactor, 23, with polymer from the first stage tubular reactor through conduits 11 and 5. The final product polymer DP is increased to within the range 40-180 or higher in tubular reactor 23 and the polymer is separated from the gas in vessel 24 with the gas passing through conduit 2 to vessel 20. If necessary the gas evolved from vessel 24, exiting in conduit 2, may be cleaned of any deleterious by-products in a manner similar to that used

on the gas stream leaving **vessel 22**, before being **returned** to **vessel 20**. The polymer product is discharged from the system through conduit 7. The pressures in the tubular reactors 21 and 23 are maintained above atmospheric pressure. The preferred pressures are in the range from about 100 psia to 2500 psia, and may be different in the two reactors 21 and 23, or in the example given.

DETD In the example above, the reaction materials for the tubular reactor 23 are obtained by reacting oligomers in reactor 21 at 200 psia and 260.degree. C., prepared from reaction material composed essentially of EG and TA entering through conduit 1, to a number average DP of 9. The oligomers exit reactor 21 through conduit 4, which is an extension of tubular reactor 21, pass into a separation vessel 22, through conduit 5, then into tubular reactor 23. The gaseous medium and gaseous by-products from the reaction in tubular reactor 23 enter the separation vessel 24, which operates at a lower pressure than reactor 23. If required, the gaseous stream exiting vessel 24 may be cleaned by employing a vessel (not shown) which is similar to gas clean-up unit 25, before **recycling** the gaseous stream through line 2 to **vessel 20**.

DETD In the embodiment of the process described as part of the example above the flow of the gaseous medium is co-current with the melt polymer or oligomer phase at all times, both within and between stages. Overall the gas enters with the reaction materials and passes through the successive stages before exiting the final **reactor** and being **recycled**.

=> d 112 6 hit

L12 ANSWER 6 OF 8 USPATFULL on STN

SUMM Applicants are not aware of any prior disclosures of adding vapor of compounds having reactive OH groups to the effluent gas to produce linear polyesters of high molecular weights that will yield gel-free, high clarity films. In fact, most of the literature advises that moisture should be kept to a minimum to prevent loss of solid-state polymerization rate. There are a few patents that mention use of water/alcohols during solid-state polymerization, but they are irrelevant to the present invention, as explained below. Treatment of poly(ethylene terephthalate) polymers with **supercritical carbon dioxide** and water under high pressure to reduce acetaldehyde is known. However, both carbon dioxide and high pressure are required and it was not under solid-state polymerization conditions. Swiss Patent Application No. 655,938 discloses a process of two stages: (1) treating the polyester with alcohol or alcohol/water until the aldehyde content is below 35 ppm then (2) postcondensing at 200.degree.-245.degree. C. It requires the measurement for acetaldehyde at the first stage and is therefore irrelevant to our present invention which applies to polymers without the need of intermediate measurement of acetaldehyde. Japanese Patent No. 59219328 discloses a process to perform moisture conditioning with a moisture content of at least 0.2 wt % to reduce acetaldehyde. The level of water disclosed is much higher than that which our present invention requires and is therefore irrelevant. Japanese Patent No. 55013715 discloses extraction of polyesters before or after solid-state polymerization by dipping the polyesters in solvents. European Patent Application No. 389,948 discloses bringing PET having an intrinsic viscosity of at least 0.50 dl/g and a density of 1.38 or more into contact with water to reduce the amounts of oligomers and acetaldehyde formed at the time of molding.

SUMM The point of introduction of the vapor can be anywhere along the process of a **continuous solid-state polymerization**. That is,

the point of introduction can be at the bottom of the reactor, close to the top of the reactor or any point in between in a continuous process. It can even be at the end of the solid-state polymerization by treating the particles with vapor in a reactor at the end of the solid-state polymerization process. It can also be anywhere along the reaction profile in a batch process. That is, vapor can be added at the beginning, towards the end or anywhere in between in a batch process.

DETD Polyester precursor pellets having an I.V. of 0.626 are continuously fed into the top of a continuous reactor and removed from the bottom in a manner such that the pellets form a slowly moving bed in which the pellets have a residence time of 54 hours. The size of the reactor is 12 feet in height and 2 feet in diameter. Temperature of the pellets entering the top is 210.degree. C. and temperature of the pellets being removed is 220.degree. C. Nitrogen is caused to enter the reactor near the bottom through a circumferential supply ring, and is removed from the top through a conduit. The nitrogen is **recycled** in conventional manner. The nitrogen temperature entering the **reactor** is 220.degree. C. and the nitrogen temperature leaving the reactor is 214.degree. C. The flow rate is 23 scfh. Water vapor at a temperature of 160.degree. C. is injected into the nitrogen stream prior to entering the supply ring in an amount such that the concentration of water vapor is 1050 ppm by weight based on the weight of nitrogen. Pellets being removed from the reactor have an I.V. of 0.93 dl/g. These pellets are subsequently extruded, into a 10 mil film, using a conventional extruder and found to be substantially gel-free by visual inspection.

=> log y

COST IN U.S. DOLLARS

SINCE FILE	TOTAL
ENTRY	SESSION
60.94	213.69

FULL ESTIMATED COST

DISCOUNT AMOUNTS (FOR QUALIFYING ACCOUNTS)

SINCE FILE	TOTAL
ENTRY	SESSION
-0.65	-35.15

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STN INTERNATIONAL LOGOFF AT 18:27:51 ON 01 DEC 2003